NWRI CONTRIBUTION 89-100

RESEARCH AND APPLICATIONS BRANCH
ANNUAL STUDY REPORT

1988 - 1989

91-05217/

FORWARD

I am pleased to present an Annual Study Report on the work of the Research and Applications Branch, National Water Research Institute.

The Scientists and Technologists of the Branch join with me in recognizing the support and stimulation provided by our many partners in these Research and Development initiatives.

We look forward to your comments and suggestions as we take on new challenges in our three special areas:

Analytical Chemistry Research
Quality Assurance
Hydraulics Studies

John Lawrence
Director
Research and Applications Branch

RESEARCH AND APPLICATIONS BRANCH

SIX-MONTH PROGRESS REPORT

1988-1989

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RESEARCH AND APPLICATIONS BRANCH

John Lawrence, Director

OVERVIEW

The Research and Applications Branch of the National Water Research Institute conducts mission oriented basic and applied research in areas of analytical chemistry and hydraulics. It is also responsible for transferring research and technology to the operational sectors of the Department and for providing technical and scientific services such as current meter calibration, sedimentology and geotechnical services and analytical quality assurance. The work addresses priority issues of Conservation and Protection and other agencies of the Department.

New, improved and more cost effective analytical techniques (including laboratory automation) are developed and evaluated for the measurement of priority chemical and biochemical parameters in environmental matrices. The Branch also has lead responsibility for analytical quality assurance for most C&P chemical monitoring programs. The major clients for this analytical work include the Institute and the Conservation and Protection National and Regional laboratories. This work is conducted in support of the Federal/Provincial Water Quality Agreements, the Prairie Provinces Water Board, the Long Range Transport of Airborne Pollutants program, and the Great Lakes Water Quality Program.

New knowledge of the dynamics of water is sought to provide a framework for the management of water resources, and for biology and chemistry research in the aquatic environment. The interfaces between water and the atmosphere, bottom sediments, and shore are studied to explain the mechanisms controlling the transport of pollutants and erosion. Advances are made in river metrology to improve management of that resource. The major clients are the Water Resources Branch, Water Planning and Management Branch, Great Lakes Water Quality Program, Environmental Protection, and the Atmospheric Environment Service.

Universities and private industry are encouraged to make use of the Branch facilities and expertise either through collaborative studies with the professional staff or leasing of the facility with or without technical support. The Branch maintains a well equipped hydraulics laboratory, sedimentology and geotechnical laboratories and a special clean and hazardous chemicals laboratory in addition to conventional chemical laboratories. All direct requests from external agencies are cost recovered, in full or in part, in accordance with the Treasury Board Cost Recovery Policy.

PROJECT OVERVIEWS - HIGHLIGHTS

ANALYTICAL CHEMISTRY RESEARCH

I. Sekerka, Project Chief

INTRODUCTION

The mandate of the project is to advance knowledge and provide expertise on environmental analytical chemistry. The project is essential for the successful management and utilization of water resources and for research on water pollution, especially the identification of emerging problems. State-of-the-art analytical methodology and techniques are one of the cornerstones for the implementation of the "Environment Economy and Sustainable Development" initiatives.

Successful solutions to environmental problems require the availability of accurate, precise and rapidly-produced analytical data. Every department program which relies on the collection of data is dependent on the availability of sound analytical methodology.

This project develops and evaluates analytical methodologies (including sample collection, preservation, extraction, pre-concentration, clean-up and analysis) for the measurement of chemical parameters in water, sediment, effluents and biota. Improvements to existing methods in terms of precision, accuracy, detection limits, cost effectiveness, automation and speciation are an integral part of the program.

Analytical methods are developed for organic and inorganic parameters in a variety of environmental substrates. These include state-of-the art techniques such as high performance gas chromatography, high pressure liquid chromatography, emission and absorption spectroscopy, flow-injection analysis, radioimmunoassay and electrochemical techniques. Both broad screening techniques and detailed quantitative techniques are developed.

Project activities include advanced analytical chemistry research as well as methodology suport and transfer to IWD laboratories, NWRI scientists, the GLWQP and other clients.

The project objectives are as follows:

a) To develop new and improved analytical methods as well as screening and sampling procedures which are accurate, and cost-effective, sensitive and unambiguous for the identification and quantification of contaminants in aquatic ecosystems.

b) To play the lead role in documentation, validation and standardization of analytical methodologies to ensure accuracy and reliability of analytical data.

c) To provide a service of sophisticated instrumentation and facilities such as gas chromatography-mass spectrometry and the Clean and Hazardous Chemical Laboratory.

d) To transfer developed methods and technologies to the national and regional laboratories of IWD and to other clients.

The current activites of the Analytical Chemistry Research Group are as follows:

Analytical/Methodological

a) Implement dense-gas (supercritical fluid) chromatography in tandem with low-resolution mass spectrometry for analyzing priority organic contaminants.
b) Develop electrochemical and flow-injection analytical methods to improve economy and efficiency of operations in WQB laboratories.
c) Develop screening methods for interference in sediment and large-volume water concentrates prior to G.C. and capillary G.C. analysis.
d) Develop clean-up procedures suitable for analysis of dioxins, furans and sulphur-containing compounds.
e) Develop a method for simultaneous determination of ions by ion chromatography.
f) Develop methods for priority contaminants employing GC-MS techniques.
g) Develop a broad spectrum analysis system for organic pollutants.
h) Development of Electrokinetic and Enzymatic Methods for Toxic and Mutagenic Effects.

Laboratory Service

a) Provide the service of the Clean and Hazardous Chemical Laboratory.
b) Automate and computerize laboratory analytical systems for application in NWQL.

Field Samplers

a) Develop, test and evaluate large-sample extractor for field use.

RESEARCH HIGHLIGHTS

A higher degree of objectivity and more reliable results of non-target compounds analysis has been achieved by using the congener method and dual column gas chromatography.

GC-MSD methods for the determination of chlorinated phenolic compounds were modified and validated for the analysis of pulp and paper industry samples.

An ion-chromatographic method for simultaneous determination of fluoride, chloride, nitrite, nitrate, sulfate, phosphate, acetate, citrate, propionate, glyconate, butyrate, formate, sulfonate and succinate ions has been developed and is in the stage of optimization and validation.

The existing protocol for the determination of Dioxins and Furans has been modified for analysis of pulp and paper industry effluents.

The previously developed Radio-Immuno-Assay method for the detection of Dioxins is being transferred to NWQL.

A sensitive, automated method for Boron has been developed. It utilizes FIA system with an on-line boron selective resin column and colorimetric detection. The method is interference free and the detection limit is 1 ppb of B.

Two types of "Super Critical Fluid Extractors" have been designed. They are a "Dynamic SCFE" and "Closed-Circuit SCFE". The comparison of Soxhlet and SCF extraction indicates substantial time saving, better reproducibility and limitation of safety risk of SCF extraction. Over 90% of PCBs from certified sediment standard have been extracted in less than 10 minutes.

An extensive sampling program using GLSE has been conducted at several locations to validate the performance of the sampler for pulp and paper industry effluents.

QUALITY ASSURANCE PROJECT

Alfred Chau, Project Chief

INTRODUCTION

The mandate of the Quality Assurance (QA) Project is to heighten awareness with respect to the need for a coordinated quality assurance program and to ensure that analytical data generated for Environnment Canada are of good and comparable quality. The Project is essential for successful management and utilization of water resources and for research on water pollution, particularly in the areas of toxic chemicals and acid rain. Above all, the assurance of data quality enables the Department to provide authoritative, credible, scientific advice on control and remediation strategies derived from interpetation of scientific data.

Chemical and biological data are essential to all decisions made by government with respect to environmental assessment and pollution control. Biased measurements lead to errors in assessment and in subsequent control actions.In addition, the expense of data collection and interpretation makes the cost of discarding questionable data very high; therefore, data must be suitable, compatible and reliable for the intended use. A comprehensive QA program is the essential first step to achieving reliable data for environmental assessment. The long-term goal of the QA Project is to assist management to develop comprehensive quality assurance programs for the Institute and the Department.

As the lead body responsible for quality assurance, the QA group:

a) Plans, coordinates, and implements quality assurance and control programs to ensure accuracy, comparability and reliability of analytical data.
b) Develops and prepares Certified Reference Materials (water, sediment and biota) and reference materials for use in quality assurance and analytical method research and environmental assessment studies.
c) Provides a consultation service on QA to this and other Departments, Provincial and Federal agencies and to international establishments.

Project activities include developing management plans for quality assurance, advising on field, laboratory and data management quality control procedures, developing Certified Reference Materials (CRM) and designing and conducting interlaboratory comparisons studies. Quality assurance studies are conducted for federal, provincial, university and private sector laboratories producing analytical data for departmental programs. Methodology evaluation and sample preservation are also carried out in support of these studies.

The QA Project is also initiating an active marketing component in advertising its products and expertise. In this regard, the first phase of the marketing activity is to advertise more aggressively the CRMs developed and prepared by the QA group. Also as resources and time permit, papers on

QA and CRMs are presented at scientific conferences and symposiums. Seven papers were presented to international conferences during this period. A brochure describing CRMs has been designed and printed for distribution.

The QA project of NWRI is also responsible for the design, management and promotion of interlaboratory QA programs. To support these goals, a national QA management workshop was organized and held in Ottawa in conjunction with the WQB of the Inland Waters Directorate. The Workshop was videotaped and distributed to interested national and international organizations.

RESEARCH HIGHLIGHTS

This year marked the completion of the Upper Great Lakes Connecting Channel Surveillance (UGLCCS) Program which was established in 1985 as a binational study to monitor the upper connecting channels with the purpose of defining the pollution contols necessary to establish a safe environment. The Quality Management Work Group (QMWG) was appointed, with the QA Project Chief acting as chairman, to ensure that quality data was produced. The QA program that was designed for UGLCCS is a highly regarded example of QA management involving two countries, several levels of government and numerous laboratories. The work of the QMWG has heightened awareness to the advantages of, and needs for, a coordinated QA program in both environmental research and management.

The QA Project prepared two detailed integrated reports on the 13 interlaboratory studies carried out previously.

In addition, the final report of the QMWG was submitted to the Management and Activity Integration Committees of the UGLCCS program. This report is a compilation of all of the QA activities and provides recommendations on issues that arose in the UGLCCS QA program but which are relevant to other multi-agency,international surveillance programs. This report will be part of the UGLCCS report.

Considerable time was also spent on providing advice and reviewing the QA components of all the final geographical reports generated for the UGLCCS.

Two large interlaboratory QA studies were finalized in the Great Lakes Water Quality (GLWQ) Surveillance QA Program. These were metals in sediments and total phosphorus in water. Two other QA studies, organics in solutions and organics in sediments are in progress. The GLWQ Surveillance QA Program is designed to ensure the quality and compatibility of data generated by both American and Canadian agencies who monitor water quality in the Great Lakes. The QA program has improved the quality of data produced by the laboratories involved in the GLWQ Surveillance program.

In addition to these two international QA programs, the QA Project has programs ongoing for three national programs. The Federal/Provincial Agreement (FPA) QA Program was created in 1986 to ensure that data generated by Environment Canada and provincial laboratories involved in the Federal/

Provincial Agreements are of good and comparable quality. The Prairie Province Water Board (PPWB) QA Program (initiated in 1982) was established to improve the compatability of provincial and federal water quality data collected from interprovincial and international streams in the prairie provinces. The data that is generated in both programs is compiled in NAQUADAT which is used extensively by a large number of users. For these two programs, two interlaboratory QA studies were sent out every month. Six data summaries and six final reports were sent to participants. For the Prairie Province Water Board, a large integrated report on laboratory performance during the year was also generated.

For the Long Range Transport of Airborne Pollutants (LRTAP) QA Program, three large QA studies for major ions and nutrients were designed and conducted. Six QA reports (two per study) were generated. In addition, computer programs were developed to provide information on laboratory performance and data quality for any given laboratory over a given time period. This is the first time such information can be provided quickly to managers and data users on data quality.

Data quality in the laboratories of the FPA, PPWB and LRTAP programs has generally improved and data quality problems have been identified for corrective action. These QA programs have a continual positive impact on data quality.

This year, there are several new key QA studies funded by outside agencies.

Eulerian Model QA Study. The new study is to design and conduct special interlaboratory QA studies to evaluate the quality and comparability of data collected in four large precipitation networks over the next two years by the governments of Canada, U.S. and Ontario and the Electric Power Research Institute (US) to validate the Eulerian Model which is being developed by Canada, West Germany and the United States.

Special Interlaboratory QA Study for Assessment of the PCB Fire at St. Basile-Le-Grand. Selected soil samples collected around the fire site were processed and used in an interlaboratory QA study designed and conducted by the QA group of NWRI to establish comparability among private, provincial and federal laboratories generating data for environmental assessment of the area.

QA Studies for Ocean Dumping Program. A study for metals in sediments was designed and conducted for EP to provide information on data quality.

Interlaboratory Study of Vegetation. This study is to assist Agriculture Canada (CFS) to evaluate QA data of an interlaboratory study on vegetation.

Development of "Audit Samples". This study funded by US EPA involves the development, characterization and archiving of six 800 L natural water samples as reference samples for laboratory auditing for future US acid rain projects.

Dioxin QA Program for the Canadian Pulp and Paper National Monitoring Program. Upon request from the National Dioxin Task Force, the Project Chief of QA formed and chaired a Quality Assurance Committee comprised of industry and federal representative to advise the Canadian Pulp and Paper Association (CPPA) and DOE management on the adequacy of procedures and on the data quality of dioxin and furan analysis of samples related to the pulp and paper industry. As a result, a detailed QA/QC protocol for dioxins and furans in pulp and paper matrices has been developed. Also, four sets of data generated by a contract laboratory have been reviewed.

Technical Transfer and Consultation

The QA group provides a leading role on QA matters and acts as Chairman or appointed members of multi-agencies, national and international bodies such as the QA Committee of the Association of Official Analytical Chemists (AOAC), the Dioxin QA Committee of the DOE Dioxin Task Force, the Quality Management Workgroup of the UGLCCS Program, the Data Quality Work Group of the IJC and the Canadian Advisory Committee on QA Management of the International Standards Organization (ISO).

Advice and consultation on QA/QC was provided for numerous environmental programs including the Federal/Provincial Water Quality Agreement, the Canadian/P.E.I. Water Resources Management Joint Study, the Prairie Province Water Board Monitoring Program and the Australian Analytical QA in Marine Quality Assessment Program.

HYDRAULICS STUDIES PROJECT

Michael Skafel, Project Chief

INTRODUCTION

The physical dynamics of water and its interaction with air, sediments, shores and man-made structures are the focus of the Hydraulics Project. Research studies on wave mechanics, river flow and sediment transport provide new scientific knowledge for use by water resource managers and engineers in Environment Canada and elsewhere. Special attention is given to understanding hydraulic processes that could lead to structural failure with serious environmental consequences.

The research program in wave mechanics creates new knowledge on the accurate parameterization of fluxes of mass, momentum, energy and heat across the air/water interface. This work has direct consequences in understanding toxic gas transfer, diffusion of pollutants, wave prediction, lake circulation, seasonal thermocline development, weather forecasting and climatic change.

The coastal engineering program is focussed on advancing the understanding of wave mechanics and sediment transport in the coastal zone, such as the resuspension of cohesive sediment, prediction techniques to develop wave climate models for sediment resuspension studies, shoreline evolution, and wave-structure interaction.

The fluvial engineering study is directed at improved understanding of fluid mechanics, sediment transport in rivers, the development of new and improved techniques for monitoring river processes, and flow-structure interaction with special emphasis on cases where structural failure could lead to serious environmental consquences. Emphasis is placed on providing information for operational branches in Environment Canada. Results are often applicable to issues in other departments and to other water resources managers.

The Hydraulics Studies Project operates the Hydraulics and Sedimentology Laboratories and provides technical support to Institute scientists and engineers using these facilities. The Project also encompasses the National Calibration Service, charged with current meter repair and calibration, primarily for the Water Survey of Canada.

A lead role is taken by Project staff in technology transfer to other government departments, universities, and the private sector. The facilities of the laboratory, when not committed to Institute activities, are actively marketed so that these other agencies have the opportunity to use our facilities as a resource in their activities. The client projects undertaken are primarily in the realms of fluvial and coastal engineering, and in the calibration of current meters.

RESEARCH HIGHLIGHTS

Sand bed streams present problems in determining flow discharge. To address such a problem on the Milk River, a flat-vee weir was selected and tested in the laboratory for the Water Resources Branch. It was found to be satisfactory in all but the most severe conditions.

An acoustic flowmeter proposed for use in remote areas (AFFRA) by the Water Resources Branch was evaluated in the towing tank. A report summarizing these tests and field tests conducted by WRB personnel was completed.

The field data collected in 1987 to examine the joint probability distribution of wave height and period has been analyzed, revealing the effects of shoaling. Work has begun on experiments in the wind-wave flume to examine the role of beach slope on the distribution.

Significant advances were made in developing our capability to examine wave directional spectra, using an iterative version of the Maximum Likelihood Method. We are well positioned to complete the analysis of wave data collected during the WAVES experiments over the past several years. Detailed cross-calibration of drag sphere and acoustic current meters has been completed, a prerequisite to the analysis of the flow data also collected during the WAVES experiment.

The work up to the Surface Wave Dynamics Experiment (SWADE) has proceeded well. Detailed plans are in place. A three metre buoy, fully instrumented, was deployed in the Gulf of Mexico, to permit calibration of the buoy systems against comparable tower mounted ones.

The gas transfer flume has been built and commissioned in the Hydraulics Laboratory. It will be used for studies of the transfer of toxics between the air and water. It has been designed so that the physical parameters (wind speed, wave conditions, turbulence) can be controlled and monitored with precision to elucidate the mechanisms controlling the transfer of toxics.

Several jobs were completed for outside clients, including physical models of a sedimentation basin, a Parshall flume, and a harbour.

STUDY REPORTS

ANALYTICAL CHEMISTRY RESEARCH PROJECT

Study 84008: Method Development for Trace Metals

by

V. Cheam, I. Sekerka, and J. Lechner

INTRODUCTION

The purpose of the study is to compare and evaluate the effectiveness of three different, sensitive analytical methods for determining trace metals in water. The three methods are anodic stripping voltametry, the new tungsten furnace atomic spectrometry and ion chromatography.

RESULTS

The study has been put on hold as the priorty has shifted to the analysis of toxic metals in pulp and paper effluents.

A literature search on this new topic has been initiated and so far indications are that very little work has been published on trace metals in paper mill effluents. The application of laser induced atomic ionization spectroscopy has been investigated.

Publications

Cheam, V., A.S.Y. Chau and W. Horn. 1988. National Interlaboratory Quality Control Study No. 35 - Trace Metals in Sediments. Inland Waters Directorate, Report Series No. 76, 61 pp.

Cheam, V., K.I. Aspila and A.S.Y. Chau. 1988. Analytical Reference Materials. VIII. Development and Certification of a New Great Lakes Sediment Reference Material for Eight Trace Metals. NWRI Contribution 88-71. Sci. of Total Environment (in press).

Cheam, V., and E Xue Li. 1988. Ion Exchange-Spectrophotometric Determination of ppb and sub-ppb Cd. Am. Chem. Soc., Div. Environ. Chem. 28(2):416-420.

Presentations

Cheam, V. Great Lakes Sediment Reference Materials for Trace Metals. Presented at the International Conference - Trace Metals in Lakes, McMaster University, Hamilton, Ontario, August 14-18, 1988.

Study 84011: Supercritical Fluid Extraction of Organics and Optimization of Separation of Pesticides by Programmed Temperature GC

by

F.I. Onuska and K. Terry

INTRODUCTION

Supercritical fluid extraction (SFE) is a separation technique based on the enhanced solvating power of supercritical fluids above their critical point. Until recently, the use of supercritical carbon dioxide extraction has been generally related to large scale chemical processing applications for coal, asphalt, coffee decaffeination, hops extraction and fractionation of polymeric materials. In the beginning of the 1980's, the emergence of Supercritical Fluid Chromatography (SFC) as a viable analytical technique has generated considerable interest in many areas of analytical chemistry and our laboratory has contributed significantly in the field of environmental analytical chemistry.

Supercritical fluid extraction has the ability to selectively extract many pollutants that are thermally labile or of high molecular weight. Since density changes of the supercritical phase are principal factors in achieving high solubility and selectivity, the role of temperature in regulating solvating power is important. Similarly, pressure changes at constant temperature can affect a dense gas's solvating power. Thus, by regulating both temperature and pressure, highly selective behaviour of supercritical fluids can be attained.

RESULTS

SFE Apparatus

Our high priority has been to design a suitable supercritical fluid extractor. In general, a supercritical fluid extraction system consists of a high pressure pumping system, an extraction cartridge and a source of supercritical fluid. We designed two extraction systems.

The first one is a dynamic extractor. Supercritical fluid flows through the extraction cartridge for up to 30 minutes and the extract is collected either into the n-pentane or methylene chloride solution or accumulated on a pre-column for GC or GC/MS analysis.

The second system operates in a close-circuit mode; assuming that an equilibrium is reached, the extraction is performed at the steady-state condition. This system provides more reproducible data.

Study 84011 (continued)

We have been using a Lee Scientific 501 model pump and various supercritical fluids such as carbon dioxide, nitrous oxide, sulfur hexafluoride and CO_2 + 2% MeOH. The sediment sample was weighed into a 0.5 mL extraction cell constructed of stainless steel. Supercritical parameters were maintained inside the extraction cell by restricting the outlet flow with the use of a 25 or 30 µm I.D. and 15 cm long fused silica tubing. The extract was collected by inserting the restrictor into a collection vial, containing for example n-hexane spiked with an internal standard (d - 10 pyrene; 200 ng/mL) or directly introduced on a pre-column of an open tubular column using GC/MS.

The comparison of Soxhlet extraction data vs SFE data indicate substantial time-saving for the SFE (30 minutes vs 8 hours), better reproducibility for extracting tetrachloro-dibenzo-p-dioxins from the spiked sediment and minimized safety risks associated with large quantities of solvents during Soxhlet extraction.

During the second half of the FY 1988/89, supercritical fluid work focussed on the utilization of the electron capture detector in SFC. Experiments were conducted to evaluate the effects of pressure, temperature and amounts of sediment required for the analysis and extraction of polychlorinated biphenyls using both static and dynamic extraction techniques. The extraction of the PCB contaminated, certified sediment standard, EC-1, obtained from the Quality Assurance Project, National Water Research Institute, was employed. Supercritical fluid carbon dixode with and without 2% methanol was evaluated at pressures of 100 and 200 atm and temperatures of 40° and 60°C. Results indicated that the static extraction of a 100 mg sample of EC-1, containing 2.02 mg/kg PCBs, with carbon dioxide and 2% methanol at 200 atm and 40°C, removed at least 90% of the PCBs in less than 10 minutes. This work has been accepted for publication in the Journal of High Resolution Chromatography.

Further, an advance has been achieved in the development of the cryofocusing technique, which enables the supercritical fluid extraction to be performed in tandem with capillary gas chromatography. The technique reduces the large volume of an extract to be trapped on the accumulator and later thermally desorbed into a capillary column. There is no contact between the analyst and a sample, and losses due to manipulation of the extract are eliminated. This technique, however, still needs some refinement, especially when very volatile components are present in the extract.

Study 84011 (continued)

Pesticide Separation

An off-line system for the generation of isothermal data based on calculation of retention indices of various pesticides has been set up. However, the available software has many bugs which must be removed by the vendor.

Publications

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Study 84011 (continued)

Presentations

Onuska, F.I. and K.A. Terry. Supercritical Fluid Extractions of 2,3,7,8-TCDD. Presented at the 3rd Chemical Congress of North American, Toronto, Ontario, June 5-11, May, 1988.

Onuska, F.I. and K.A. Terry. Supercritical Fluid Extraction: Trials, Tribulations and Trends. Presented at COLACRO-2, the 2nd Latin-American Congress in Chromatography, Buenos Aires, Argentina, October 1988.

Study 84012: Preconcentration for Ultra-Trace Analyses

by

D.H.J. Anthony

INTRODUCTION

Research conducted under Study 84012 during FY 87/88 led to the development of two working versions of large-sample extraction systems (one to provide a 24 hour integrated extract and one to provide a one hour integrated extract) as well as a third prototype system designed to investigate the feasibility of weekly-integrated extraction. This technology is collectively referred to as Goulden Large-Sample Extraction technology.

The 24 h version of the extractor was accepted by the four parties conducting the Niagara River Toxic Management Plan as a validated "state-of-the-art" preconcentration method for the ultra-trace determination of organic contaminants.

The technology described was designed to process relatively "clean" waters (low DOC, POC) in response to requirements of the WQB (NWQL and WQB-OR) in determining organic contaminant levels in the Niagara River and Great Lakes systems.

The general objectives of this study during the 1988/89 fiscal period are to investigate the application of GLSE technology to the processing of aqueous matrices other than the type mentioned above such as humic waters, water receiving pulp and paper mill effluents, estuarine waters, precipitation and to further develop the technology to improve its use in routine and research applications by incorporation of features such as solvent recovery, isolated operation and unattended operation.

RESULTS

During the periods 08-17 June and 08-19 August, 1988, a study was made in collaboration with the WQB-OR, of the application of GLSE technology in the determination of organic contaminant levels in waters receiving pulp and paper mill effluents. The study was conducted on the Rainy River system at Fort Francis, Ontario, the site of two Boise (Carcade) pulp and paper mills. Sampling was carried out at six locations (upstream control site, downstream site, plume sites for each mill and sites within each mill sampling effluent before dilution by the river). Separate extractions were carried out for a "standard" suite of contaminants (these included in the NRTMP) and for dioxins. Both clarified and whole water samples were processed for the determination of dioxins.

One-hundred litre water samples were processed using GLSE, at selected Rainy River locations, and one at Niagara-on-the-Lake, for studies in the RIA determination of 2,3,7,8-TCDD.

Study 84012 (continued)

During the period October 03-24, 1988, the application of large-sample extraction systems to the preconcentration of trace organic contaminants in the St. Lawrence River system was investigated onboard the CSS LIMNOS (Cruise #88-07-01). The performance of these systems in the processing of estuarine waters was of particular interest.

Extractions were carried out on clarified (Westfalia centrifuge) water at twelve stations in the river, six in the riverine lake section between Kingston and Quebec City and six in the Upper Estuary between Quebec City and Tadoussac, Quebec.

At each station, centrifugation periods were adjusted to collect sufficient particulates for the various analyses required (2-4 hours). Over each centrifugation period, 100 L of centrifuged water were extracted using the Goulden Large-Sample Extractor (GLSE) at a nominal sampling rate of 1 L/min. The total time spent sampling at each station was variable (12-48 hours) with centriguation and extraction being carried out continuously. A total of 80 extracts were collected during this cruise. As part of the cruise, four days were spent in Quebec City, coinciding with the "International Symposium on the Fate and Effects of Toxic Chemicals in Large Rivers and Their Estuaries". During this time, operation of the large-sample extractors was described to conference attendees and dignitaries as part of an "Open House" onboard the CSS LIMNOS.

A student, hired under the COSE program, was supervised in the design and construction of a prototype microprocessor-based controller dedicated to the control of various operating features of the GLSE systems. The suitability of this device, and other commercially available devices, in providing this control will be tested during the balance of this fiscal year.

Discussions with client (C.H. Chan, WQB-OR) and researchers in the field of precipitation sampling has led to the conclusion that GLSE technology would be inappropriate for use in the existing precipitation sampling methodology as it would add unnecessary complexity to a system which is performing adequately. This segment of the study will not be pursued further.

Presentations

Anthony, D.H.J. Application of Continuous Flow Liquid-Liquid Extraction Technology to the Great Lakes/Niagara River Toxic Organic Contaminant Monitoring Programs. Presented at the 31st Conference of the International Association for Great Lakes Research, McMaster University, Hamilton, Ontario, May 17-20, 1988.

Anthony, D.H.J. Large-Sample Solvent Extraction Technology for Improving the Detectability of Organic Contaminants in Water. Presented at the 3rd Chemical Congress of North America, Toronto, Ontario, June 5-11, 1988.

Study 84013: Continuous Flow Analytical Systems Research

by

I. Sekerka and J.F. Lechner

INTRODUCTION

There are two separate parts of this study; to develop analytical methods as required by water quality assessment and to optimize the effectiveness and economy of environmental laboratories and environmental monitoring.

The results of our previous research proved that the "Flow Injection System" is capable of improving both goals. FIA methods for Mg, Ca, Ba, Na, K, NH_4, NO_3^-, NO_2^-, PO_4^{3-}, CN^-, alkalinity and acidity are now used on a routine basis.

The problem of presenting samples to the FIA system (up to 300 samples per hour) led to the development of an automated robot system. In the final version, it will handle entire strings of operations required for the analysis (sample identification, delivery to the analyzer, operation of the analyzer, data collection, calculation of the results, statistical evaluation and storage of the data).

RESULTS

A sensitive, automated method for the determination of Boron, as requested by NWQL, has been developed. It is a "Flow Injection" method using an on-line preconcentration column of boron selective Amberlite IRA 743 resin. Boron from the sample is accumulated in the column for a preselected period of time and then eluted and mixed with colour forming reagent and the intensity of the colour is measured spectrophotometrically and related to the concentration of boron in the sample. The method is free from interference, including that resulting from the original colour of the sample. The lower detection limit is a variable parameter. The longer the time of accumulation, the lower the detection limit. At a sample rate of 20 samples/hr, the detection limit is 5 ppb, whereas at a rate of 10 samples/hr, the detection limit is 1 ppb.

The development of the robotic system continued with modifications made to both the hardware and software. Extensive tests of the robotized FIA-ICAP system under the condition of routine daily operation in NWQL proved very good performance indicators in terms of reproducibility, reliability and time effectiveness.

Study 84013 (continued)

Publications

Sekerka, I. and J.F. Lechner. 1988. Determination of Alkalinity of Water by FIA with Conductometric Detection. NWRI Contribution 88-68.

Sekerka, I., J.S. Ford, J.F. Lechner and Y.M. Sheikh. 1988. Determination of Acid Neutralizing Capacity of Water and Related Problems. NWRI Contribution 88-103. Int. J. of Environmen. Anal. Chem. (in press).

Sekerka, I. and J.F. Lechner. 1989. Automated Method for the Determinaton of Boron in Water. NWRI Contribution 89-83. Analyst (in press).

Sekerka, I. and J.F. Lechner. 1989. Instruction Manual for Determination of Ca and Mg by Flow Detection Analysis and Atomic Absorption Spectroscopy. NWRI Contribution 89-84.

Study 84014: Analysis for Organics in Dense Matrices

by

B.F. Scott

INTRODUCTION

During the gas chromatographic analysis of a processed organic extract from the aquatic environment, the target compounds usually comprise only a small fraction of all the detected compounds. As part of a study which will parameterize the non-target compounds, the identification and quantitation of polychlorinated biphenyls (PCBs) was undertaken. These compounds are recovered in the hexane eluate fraction of the cleanup as are chlorinated benzenes and several organochlorines (OCs). At present, the hexane eluate is split with single packed column gas chromatography being used to estimate the concentration of PCBs, and dual capillary column gas chromatography being used to determine the concentration of organochlorines. The present work involved identifying and quantifying each contributing PCB compound in a sample as well as the organochlorines separated by dual capillary column gas chromatography.

EXPERIMENTS

The first task was to prepare a standard solution from standard PCB mixtures that are commercially prepared. There is a total of 209 possible PCB compounds or congeners. (Here congeners are individual members of a particular structural type which can vary in the degree of substitution.) In the chromatographic standard there were about 110 congeners, ranging from monosubstituted to fully substituted decachlorobiphenyl. Using a dual column mode, the linear range for detection was from 40 to 5000 pg/μL total PCB concentration injected. Also the reproducibility as measured by the coefficient of variance was less and 10% for each congener on both columns over the concentration range studied. The PCB standard was then chromatographed with the organochlorine standard to check for possible interference. Several interferences were found between the PCBs and OCs on both columns, but the interference was not the same for the two columns of differing liquid phase. Therefore, by examining the output of both columns it is easily determined if a particular peak is a PCB or OC.

This work was initiated at the request of the National Water Quality Laboratory, and the methodology was to be used in their Automated Organic Laboratory. A central computer controls up to ten gas chromatographs and collects data from a maximum of 18 detectors. As there is considerable data generated in the PCB analysis, two computer programs were created for this central computer to facilitate the data handling. First, a statistical program was written to determine the mean, standard deviation and coefficient of variance from the result files generated by the gas chromatographs. The second program was written to provide an output where the PCB congeners are grouped as to the degree of substitution and confirmed concentrations are noted when a congener is detected from both columns. The program lists

Study 84014 (continued)

co-eluting peaks, which are not necessarily the same for both columns, and the OC concentrations obtained from both columns.

It was determined that the simplest way to run the program is to select one of the columns as the working column and use the other as the confirmation column. This facilitates the analysis of samples which exhibit negative peaks which influence the baseline and hence the calculation of concentration of compounds eluted near the negative peak. Also impurities may coelute with some of the PCB congeners or OCs which will give enhanced concentrations. In both instances, the concentrations determined on the confirmation column can be substituted for those derived from the working column.

RESULTS

After the preliminary work was completed, a number of PCB mixtures were analyzed and the results, based on the confirmed presence of congeners, are shown below.

Table 1

Comparison of Total PCB Concentrations in Standards

Aroclor Standard	Expected Concentration (pg/µL injected)	Analyzed Concentration (pg/µL injected)
1221	50	58.8
1016	41	40.4
1262	50	42.9
1221 + 1254 + 1260	200	203.7
1260	200	171.2
1242	200	187.4
1254	200	182.1
Standard sediment	(50)	37.4

The original output had the individual congeners arranged by degree of substitution and the concentration of each congener. Sediment samples that had been previously analyzed by routine methods were analyzed for OCs and PCBs. Typical results are shown in Table 2.

Study 84014 (continued)

Table 2

Concentrations of Organics in Sediment Sample.
(ng/g)

Compound	Sample 1		Sample 2	
	Previous Work	This Work	Previous Work	This Work
1,2,4-Trichlorobenzene	2.30	1.90	4.94	4.98
1,2,3,4-tetrachlorobenzene	2.50	2.55	2.99	3.01
Pentachlorobenzene	2.18	2.29	3.38	3.27
HCB	6.12	0.34	1.45	1.99
p,p-DDE	3.63	3.06	3.49	4.48
Mirex	0.68	0.90		
PCB	146.4	279.5	50.6	51.4

There is good agreement for almost all the organochlorines in both samples, but the new value for the PCB concentration in Sample 1 is twice that obtained previously. There is a higher degree of objectivity using the congener method, and dual column chromatography was employed which would produce more reliable results. The results were presented at the Third Chemical Congress of North American (Toronto, June 1988) and Broad Spectrum Analysis Workshop (Regina, June 1988). While awaiting technology transfer to NWQL, the method was used to analyze several priority samples.

Several other activities were entered into. One was the PCB congener analysis of samples submitted by the Green Peace Organization. Also, a comparative analysis study on samples collected from around the Great Lakes was undertaken where the results generated from the congener methodology were compared to those obtained using standard methods. These results were reported (Scott and Onuska, 1989). In addition, a series of validation samples including sediment, spiked sediment, spiking solution, standards, and cleaned-up spiking solution were analyzed by a standard and the congener specific methodology. These solutions contained organochlorines including PCBs. The two methods gave comparative results and these are being prepared for publication.

Study 84014 (continued)

Publications

Scott, B.F., F.I. Onuska and J. Kohli. 1988. Determination of Polychlorinated Biphenyls and Organochlorines by Dual Column Gas Chromatography. NWRI Contribution 88-78.

Scott, B.F. and M. Misunis. 1988. Enhanced Gas Chromatographic Quantitation of Organochlorines. NWRI Contribution 88-111.

Scott, B.F. and F.I. Onuska. 1989. Analysis for Polychlorinated Biphenyls by Dual Capillary Column Gas Chromatography. NWRI Contribution 89-85. The Journal of Microcolumn Separations (in press).

Scott, B.F. 1988. Analysis of PCBs and Organochlorines by Dual Column Capillary Gas Chromatography. Proceedings of the 23rd Annual Workshop for Pesticide and Trace Organic Residue Analysis (Western Canada), April 27-28, Regina, Saskatchewan, p. 53.

Presentations

Scott, B.F., J. Kohli and F. Onuska. The Automated Analysis of OCs and PCBs by Capillary GC. Presented at the 3rd Chemical Congress of North America, Toronto, Ontario, June 5-11, 1988.

Scott, B.F. Analysis of PCBs by Dual Capillary Columns - Applicability to Broad Spectrum Analysis. Presented at the Broad Spectrum Analysis Meeting, Regina, Saskatchewan, June 9, 1988.

Scott, B.F. Analysis of PCBs and Organochlorines by Dual Column Capillary Gas Chromatography. Presented at the 23rd Annual Workshop for Pesticide and Trace Organic Residue Analysis (Western Canada), Regina, April 27-28, 1988, O. 53.

Study 84016: Operation of CHCL and Separation of Organics

by

I. Sekerka and R. Wilkinson

INTRODUCTION

Burgeoning interest in the formation of dioxins and furans during the bleaching process of the pulp and paper industry, has resulted in a sharp increase in the demand for the services of the Clean and Hazardous Chemical Laboratory (CHCL). Standards presently being requested by NWQL, Fisheries and Oceans and RAB now contain up to eighteen components for the GC/MS analysis of dioxins and furans.

Recent programs created to determine the source and extent of dioxin contamination with paper production have required the application of RAB methodology to the cleanup of various sample matrices such as mill effluent, sludge, pulp, sediment, and oyster tissue, to establish the applicability of the methodology and the modifications required.

The use of woodchips contaminated with pentachlorophenol, etc., in the paper-making industry, and its possible role in dioxin formation, has created an interest in determining if de-chlorination of the higher chlorinated dioxins results in the formation of the more highly toxic 2,3,7,8-tetrachloro-dibenzo-p-dioxin (2,3,7,8-TCDD).

The combustion of PCBs at a storage facility in St. Basile-Le-Grand, Quebec, and concern about possible dioxin/furan contamination of the immediate environs, required the full-scale participation of the CHCL to determine the extent and level of dioxin/furan formation and contamination.

The low solubility of dioxins and furans in aqueous media, has caused an interest in determining the applicability of the large-volume extractor for the analysis of pulp mill effluents for the presence of dioxins and furans.

RESULTS

Joint participation of the NWQL and CHCL staff in an interlab sample exchange of selected pulp and paper mill matrices, has resulted in modification of existing RAB methodologies to accommodate the unique matrices associated with the paper making process. Introduction of an acidified silica gel column greatly reduced the "background" and enhanced the analysis of such matrices. Determination of modifications to the methodology is an ongoing process.

The successful analysis of sediment samples from paper mill sites in British Columbia has enabled the ongoing participation of the CHCL, in a joint program to determine if successive dechlorination of higher

Study 84016 (continued)

chlorinated dioxins occurs, and if the formation of 2,3,7,8-TCDD is feasible by this process.

Analysis of aqueous, sediment and "wipe" samples from St. Basile-Le-Grand, Quebec, determined the extent of the contamination. Analysis of large-volume pulp mill effluent samples have indicated that the RAB methodologies can successfully clean up the samples for the determination of dioxins and furans.

Joint participation of Quality Assurance staff and CHCL staff in the analysis of a sediment sample for an interlab round-robin and possible development of a CRM for dioxin/furan analysis was completed. Also, forty samples spiked with high and low concentrations of ^{13}C surrogates were prepared for the round-robin check sample assessment.

Analysis of thirteen centrifuged particulate samples from large volumes of pulp mill aqueous effluent samples, has proven to be time consuming and fraught with difficulties (interferences). Recoveries of the ^{13}C surrogate cocktail, have been above acceptable levels (40-120%) but only after additional cleanup procedures have been applied.

Analysis of sixty samples of B.C. pulp mill sediment has indicated little or no de-chlorination activity.

A total of 95 samples of various matrices have been analyzed in support of the pulp and paper mill program.

Refurbishing of the CHCL was recently completed and the facility has been returned to full production, following a five-day shutdown.

Publications

Onuska, F.I., R.J. Wilkinson and K. Terry. 1988. Isomer-Specific Separation and Quantitation of Tetrachloro Dibenzo-p-Dioxins by HRGC and HRGC/MS. J. High Resol. Chromatogr. Chromatogr. Commun. 11:9-12.

Study 84017: Screening Techniques for Toxic Substances

by

J. Sherry and R. Wilkinson

INTRODUCTION

The timely and cost-effective analysis of trace contaminants such as polychlorinated dibenzo-p-dioxins (PCDDs), is a high priority. Conventional methods for the analysis of such contaminants tend to be time-consuming and expensive. The employment of screening tests would make possible the elimination of analyte-free samples from further analysis and thereby increase overall analytical efficiency. The radioimmunoassay for the detection of PCDDs is being developed to satisfy an IWD requirement for such a screening capability.

Immunoassays use the sensitive and highly selective binding capabilities of antibodies to detect trace contaminants in environmental samples. For some parameters, such as PCDDs, sample extraction and cleanup is necessary; for other parameters no sample preparation is required. It is hoped eventually to have a battery of short term immunoassay tests for the detection of a variety of environmental contaminants.

RESULTS

A review of progress to-date on the RIA for PCDDs was presented at the Third Chemical Congress of North America in June. The detection limit of the RIA for PCDDS was determined to be 60 ppt using fish matrix. A new synthetic route for the synthesis of an improved iodine-125 labelled dioxin derivative was explored. The RIA for the detection of PCDDs is being transferred to the NWQL. As part of this process some modifications to the currently used PCDD extraction and cleanup method are being evaluated in conjunction with NWQL staff. It is hoped that such modifications will lower the cost and improve the sensitivity of the RIA. The results for our studies into the detection of TCDD in fish were presented at the Dioxin-88 international conference.

A presentation on the RIA for PCDDs was made at the September meeting of the National Task Force on Dioxins in Pulp and Paper Mill Effluent. Several large volume effluent samples have been extracted and prepared for analysis using RIA.

Agreement has been reached with Lawrence Livermore National Laboratories on the desirability of evaluating anti dioxin monoclonal antibodies in the RIA. Such work will be of a collaborative nature.

Immunoassays for the widely used herbicide, Atrazine, are under evaluation. Initial results were present at the BIOQUAL-88 Workshop. Indications are that the detection limit of conventional techniques will be easily surpassed.

Study 84017 (continued)

Tyramine derivative of 1-amino 3,7,8-trichlorodibenzo-p-dioxin was synthesized and characterized. This hapten will be labelled with ^{125}I for use with the RIA for PCDDs. Supportive funding for this work was obtained from the NWQL (2.5K).

An improved extraction and cleanup procedure for the determination of PCDD residues in fish tissue was developed for use with the RIA for PCDDs. The method is more cost-effective and efficient than the previous method; all liquid/liquid steps have been eliminated. The method was statistically validated at high and low spiking levels (20 ppt and 100 ppt).

Tube and microtitre plate versions of the immunoassays for Atrazine were evaluated using spiked river and lake water. A variety of raw samples and sample extracts were analyzed using the immunoassay and conventional techniques. The choice of immunoassay will depend on operational requirements: the tube and plate versions are more suitable for field and laboratory use respectively.

Publications

Sherry, J.P., J. ApSimon, L. Collier, B.K. Afghan, and P. Albro. 1988. Radioimmunoassay for the Detection of Polychlorinated Dibenzo-p-dioxins in Environmental Samples: Method Description. NWRI Contribution 88-73.

Sherry, J.P. J. ApSimon, L. Collier, R. Wilkinson, P. Albro, B. Afghan. 1988. The Use of Radioimmunoassay for the Detection of Polychlorinated Dibenzo-p-dioxins in Fish Samples. NWRI Contribution 88-76. Chemosphere (in press).

Sherry, J.P. 1989. A Comparison of the Abilities of 15 Media to Recover Viable Yeasts from Pond, River, and Lake Water. Journal of Microbiological Methods (in press).

Sherry, J.P., B.K. Afghan, J. ApSimon, L. Collier, F. Bishai and P.W. Albro. Radioimmunoassay for the Detection of Polychlorinated Dibenzo-p-Dioxins in Environmental Samples: Introduction, Preliminary Evaluation and Development of a Working Assay. NWRI Contribution 89-80.

Presentations

Sherry, J.P., J.A. ApSimon, L. Collier, R.J. Wilkinson, P.W. Albro and B.K. Afghan. Detection of Polychlorinated Dibenzo-p-dioxins in Fish Using Radioimmunoassay. Presented at the 3rd Chemical Congress of North America, Analytical Program, Toronto, Ontario, June 5-10, 1988.

Study 84017 (continued)

Sherry, J.P. The Use of Radioimmunoassay for the Detection of Dioxins in Environmental Samples. Presented at the 4th Workshop on Dioxin/Furan Emmissions from Pulp and Paper Mills, Burlington, Ontario, September 7 and 8, 1988.

Sherry, J., J. ApSimon, L. Collier, R. Wilkinson, P. Albro and B.K. Afghan. The Use of Radioimmunoassay for the Detection of Polychlorinated Dibenzo-p-dioxins in Fish Samples. Presented at Dioxin '88, the 8th International Symposium on Chlorinated Dioxins and Related Compounds, Umea, Sweden-ANA P29, August 21-26, 1988.

Sherry, J.P. Detection of Contaminants in Environmental Samples Using Radioimmunoassay Techniques. Presented at Bioqual '88, Burlington, Ontario, October 18-21, 1988.

El-Shaarawi, A., J. Sherry and S.R. Esterby. Application of Intervention Analysis to the Assessment of the Response of Bacteria to Stresses. Presented at the 1st Biennial Water Quality Symposium: Microbiological Aspects, Session VIII, Banff, Alberta, Canada, August 29-September 2, 1988.

Study 84018: GC-MSD Methods Development for Priority Organics

by

H.B. Lee

INTRODUCTION

Pulp and paper effluents are known to be toxic and mutagenic to fish and other organisms. Among the toxic pollutants present in these effluents, chlorinated phenolic compounds such as guaiacols, catechols, phenols, syringols, and vanillins are collectively a major class of contaminants. Although analytical methodologies are available for such compounds, none of the existing methods are comprehensively validated. The main objective of this study is to develop a validated method suitable for the routine determination and confirmation of all major chlorinated phenolics in pulp mill effluents.

Aniline and its chlorinated and methylated derivatives as well as chloronitrobenzenes are carcinogenic compounds. They occur in the environment either as industrial wastes and metabolites or degradation products of many herbicides and pesticides. Some of these toxic chemicals are listed as priority pollutants under CEPA and US EPA. The other objective of this study is to develop sensitive and specific analytical methods for the above compounds.

RESULTS

Based on the proven in-situ acetylation technique, a method was successfully validated for a group of 31 chlorinated phenolics which are commonly found in paper mill effluents. Analysis of these acetyl derivatives were performed on a 30 m DB-5 column interfaced to an electron capture detector (ECD). For compounds with only one chlorine substitution, selected ion monitoring with a mass selective detector was employed since they were insensitive to the ECD. This method was validated at three levels of fortification, i.e., 400, 40, and 4 µg/L, using an effluent sample which was nearly free of the above phenolics. Quantitative recoveries were obtained for all phenols with the exception of chlorocatechols in a few cases. Application of this procedure to several effluent samples collected from an Ontario mill produced results which were consistent with those reported in the literature. This method is being further evaluated by the Wastewater Technology Centre for the determination of phenols in effluents. A final report was written.

In preparation for the development of analytical methodology for environmental samples, several derivatization procedures for the GC detection of the chloroanilines were examined. In view of their sensitivity, GC resolution, stability, and completensss of formation, the heptafluorobutyrl amide derivatives were found to be most suitable for the analysis of anilines in this study.

Study 84018 (continued)

EI GC-MS data for these derivatives were also collected for confirmation purposes. A final report was written and published. A method for the determination of toluidines and a few other methylated anilines in water was also developed. This method involved the formation of pentafluoropropionyl amide derivatives of the anilines and GC-ECD and GC-MSD analyses and had a detection limit of 0.05 µg/L. A final report is in preparation.

A method for the determination of 14 chloronitrobenzenes in natural water and fish samples was also developed. The method detection limits were 2 ng/L for water and 5 ng/g for fish samples. A final report was written. This method has been applied to the determination of bioconcentration factors of chloronitrobenzenes in rainbow trout.

Publications

Lee, H.B. 1988. Perfluoro and Chloro Amide Derivatives of Aniline and Chloroanilines: Their Formation and Gas Chromatographic Determination by Mass Selective and Electron-Capture Detectors. NWRI Contribution 88-66. J. Chromatogr. 457:267-278.

Lee, H.B., R. Hong-You and P. Fowlie. 1988. Chemical Derivatization Analysis of Phenols. VI. Determination and Confirmation of Chlorinated Phenolics in Pulp and Paper Effluents. NWRI Contribution 88-110.

Lee, H.B. and G.D. Kissoon. 1989. Determination and Confirmation of Chloronitrobenzenes in Water and Fish Samples. NWRI Contribution 89-81.

Lee, H.B., E.A. Kokotich and J.A. Abbott. 1988. Determination and Confirmation of Some Synthetic Pyrethroids and Their Metabolites in Water Samples. NWRI Contribution 88-70.

Niimi, A.J., H.B. Lee and G.D. Kissoon. 1989. Octanol-Water Partition Coefficients and Bioconcentration Factors of Chloronitrobenzenes in Rainbow Trout (Salmo gairdneri). Environ. Toxicol. Chem. (in press).

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Tse, H., J. Carron, M. Forbes and H.B. Lee. 1989. A Multi-Residue Method for the Determination of Chlorobenzenes, PCB, Organochlorinated Insecticides, PAH and Chlorophenols in Suspended Sediments. NWRI Contribution 89-82.

Study 84019: Method Development for Organic Acids

by

V. Cheam

INTRODUCTION

It is recognized more and more that organic acids play a very important role in atmospheric chemistry and precipitation composition. In fact, Grosjean et al, very recently reported that in the Los Angeles smogs, acetic and formic acids are present in greater amounts than nitric and hydrochloric acids (American Chemical Society, Division of Environmental Chemistry, 196th National Meeting, Los Angeles, California, September 25-30, 1988).

In routine analysis, ion chromatography is used to determine the common inorganic anions (Cl^-, NO_3^-, NO_2^-, SO_4^-). Fluoride cannot be routinely analyzed without using eluent matching due to the presence of "water dip" and interference from some organic acids. However, it would be advantageous to be able to determine F^- and organic acids in the same run along with the common inorganic anions.

The aim of the study is to develop an ion chromatographic method that can simultaneously analyze inorganic anions, including F^-, and mono-, di-, and tri-protic organic acids, which are resolved within a reasonably short time to be useful in routine analysis.

The study was initiated at the request of the National Water Quality Laboratory.

RESULTS

An ion chromatographic system was set up consisting of a gradient pump, a conductivity detector, an eluent delivery system, an analytical column and a data collector/processor.

Isocratically, it was impossible to achieve the desired resolution of anions. Gradient elution had to be used and was optimized using a weak eluent (2 mM NaOH) at the beginning of analysis followed by a strong eluent (90 nM NaOH). These conditions eliminate the "water dip" and, as shown in Figure 1, resolve the following ions in less than 10 minutes: F^-(1), acetate(2), propionate(3), glyconate(4), butyrate(5), formate(6), methane sulfonate(7), Cl^-(8), NO_2^-(9), succinate(10), SO_4^{2-}(11), PO_4^{3-}(12), NO_3^-(13), citrate(14). Oxalate can also be easily resolved and elutes between the sulfate and phosphate peaks.

Study 84019 (continued)

Of the 17 acids potentially present in precipitation-related samples, hydroxymethylsulfonate coelutes with succinate, whereas lactate coelutes with acetate. However, lactate is rarely present or is present in much smaller quantity than acetate according to Keene and Galloway. Also succinate is rarely found in these types of samples.

Three different types of water samples were used for recovery studies, which resulted in percent recoveries of 100 ± 10%.

The method can be readily adapted for routine use. A final report has been completed.

Publications

Cheam, V. and E Xue Li. 1988. Ion Exchange-Spectrophotometric Determination of ppb and sub-ppb Cd. Am. Chem. Soc., Div. Envir. Chem. 28(2): 416-420.

Cheam, V. and E Xue Li. 1988. Ion Chromatographic Determination of Low Level Cd(II), Co(II), and Mn(II) in Water. NWRI Contribution 88-65. J. Chromatogr. 450:361-371.

Presentations

Cheam, V. Ion-Exchange/Spectrophotometric Determination of ppb and Sub-ppb Cd, presented at the American Chemical Society 196tn National Meeting, Los Angeles, California, September 25-30, 1988.

STUDY REPORTS

QUALITY ASSURANCE PROJECT

Study 84021: Organic Quality Assurance

by

Yvonne D. Stokker and A.S.Y. Chau

INTRODUCTION

The provision of Interlaboratory Quality Assurance studies for various organic parameters is a valuable and very necessary service for any laboratory involved in environmental management, monitoring and surveillance, or research. This Organic QA Program designs, conducts, and provides interpretation of interlaboratory QC studies in support of the Federal Interdepartmental Committee on Pesticides (FICP) and the Federal/Provincial Quality Assurance Program as well as executing special studies under the National Program. Each participating laboratory receives a summary of all interlaboratory data from which peer appraisals can be made. Precision and accuracy data can be determined for each laboratory when blind duplicates and CRMs are included among the samples distributed.

The effectiveness of interlaboratory Quality Assurance studies, intralaboratory Quality Control, and analytical methods research is strongly dependent on the availability and use of reference materials (RMs) and certified reference materials (CRMs). These may be analytical standards, either neat or in solution, sample extracts, or natural substrates such as samples of water, sediment or biota. They must be stable, well-characterized, and homogeneously subdivided into identical aliquots. Their matrices should be identical, or at least very similar, to those of the field program materials in order to eliminate or greatly reduce the matrix effect between reference and test samples. The target value for each parameter must be well established and supported by sufficient valid documentation. Each RM and CRM should be periodically monitored in order to ensure the stability of all parameters over time.

Specifically, the major goals for this year were as follows:

1) to prepare Reference Samples in ampules for organics;

2) to initiate, design, and conduct two interlaboratory studies for organics;

3) to monitor the stability of selected organic parameters in reference materials;

4) initiate the preparation of a new sediment CRM (EC-7); and

5) to communicate and market the QA products and expertise.

Study 84021 (continued)

RESULTS

The preparation of reference materials in ampules was supported this year by the PESTMYOP Program and the IJC Quality Control Program.

For PESTMYOP, two reference sediment extracts in ampules were prepared. Freeze-dried sediments from Lake Erie and Lake St. Clair were extracted, combined and concentrated so that 1 mL of the final product represented 2 g of the original freeze-dried sediments. Preliminary screening of this extract revealed the presence of approximately 100 ng/mL PCBs, 10-1000 ng/mL PAHs and 1-50 ng/mL chlorobenzenes. The extract was split, fortified to two levels with several s-triazines, including Atrazine, and then sealed into glass ampules. These ampules were then tested for homogeneity and monitored for stability of the parameters of interest. Seven mixtures of triazines in the form of standard solutions were also prepared and sealed into glass ampules. Ampules containing the sediment extracts as well as those containing the analytical reference standard solutions were shown to be homogeneous at a 95% confidence level with respect to the four s-triazines of interest. Both types of ampule mixtures proved to be stable for up to six months.

For the IJC Quality Control Program, ampules of standard solutions were prepared for Aroclor blends, chlorobenzenes, organochlorinated insecticides, neutral herbicides and s-triazine herbicides. In addition, a fish extract containing 18 PAHs at approximately 10 µg/mL was prepared, cleaned up by Gel Permeation Chromatography (GPC) and activated Silica Gel, and sealed into ampules.

National Study No. 36 on the Analysis of Aldicarb in Groundwater was conducted to meet the requests of the Water Quality Branch, Atlantic Region and the Groundwater Contaminants Group of NWRI. This interlaboratory QA study provided an exceptionally strong case to support the necessity and value of interlaboratory studies in methods research, laboratory performance assessments and in judging the comparability of data.

Three new studies were initiated this year as follows: (1) In a study being conducted for the FICP (CAPCO) Check Sample Program, 34 laboratories across Canada analyzed for 18 organochlorinated insecticides (OCs) in natural water samples. (2) Study No. 1 of the new Federal/Provincial Organic Interlaboratory QA Program was conducted with eight laboratories having received a series of injection-ready ampules containing 18 OCs in iso-octane. (3) Thirty-two laboratories received sets of ampules of standard solutions and sediment extracts containing Atrazine. This special QC study entitled National Study No. 37, was conducted as part of the PESTMYOP study on sediment reference extracts in ampules.

As part of the QA team's marketing strategies for QA and its products, a display unit was designed and used at the 1988 CCIW Open House to advertise the reference materials developed by the QA group.This display generated considerable interest in both the reference materials in ampules

Study 84021 (continued)

as well as the sediment CRMs. A major step towards marketing these QA products has now been taken by having 3000 advertising brochures printed to inform potential clients of the necessity and availability of the sediment CRMs. The brochures will be widely distributed to laboratories involved in all fields of environmental studies, from applied and investigative research to routine monitoring and surveillance.

PRELIMINARY REPORTS

Date	Report	Author
May 1988	National Interlab QC Study No. 36 on the Analysis of Aldicarb and Its Oxidative Metabolites in Groundwater	Y.D. Stokker
Dec. 1988	Quality Assurance Support in the Evaluation of Field and Labortory Protocols for the Analysis of Metals in Lake Water and Sediment.	Y.D. Stokker & H. Alkema
Jan. 1989	Preservation of Organics. Part IV. Stability of Polynuclear Aromatic Hydrocarbons in Preserved Fish Homogenates.	Y.D. Stokker
Jan. 1989	FICP Report. Interlaboratory Study on the Analysis of Organochlorinated Insecticides in Natural Waters.	Y.D. Stokker & A.S.Y. Chau
Mar. 1989	National Interlab QC Study No.37 on the Analysis of s-Triazines in Sediment Extracts.	Y.D. Stokker & A.S.Y. Chau
Mar. 1989	Fed./Prov. Organic Interlab QC Study No. 1 on the Analysis of Organochlorinated Insecticides in Ampules.	Y.D. Stokker
Mar. 1989	PESTMYOP Report on the Preparation and Stability of Sediment Reference Extracts in Ampules.	Y.D. Stokker & J.E. Milne

Study 84021 (continued)

Publications

Stokker, Y.D. and A.S.Y. Chau. 1988. Summary FICP Report. Interlaboratory Study on the Analysis of Chlorophenols in Natural Waters. NWRI Contribution 88-112.

Lesage, S. and Y.D. Stokker. 1988. Aldicarb Residues in Water: A Perspective. Proceedings of the XXth Annual Eastern Canada Workshop on Pesticide Residues and Environmental Contaminants, June 1988, Sault-Ste Marie, Ont.

Stokker, Y.D. 1988. Method for the Analysis of Acid and Neutral Herbicides in Sediments. NWRI Contribution 88-67.

Li, W.C., H.B. Lee and A.S.Y. Chau. 1988. Organic Standards for Preparation of Blind Spikes. NWRI Contribution 88-64.

Afghan, B.K. and A.S.Y. Chau [Eds.]. 1989. Analysis of Trace Organics in the Aquatic Environment. CRC Press Inc., Boca Raton, Fla., U.S.A. 346 pp.

Presentations

Chau, A.S.Y. Development of the World's First Sediment Certified Reference Materials. Presented at the 3rd Chemical Congress of North America, Toronto, Ontario, June 5-11, 1988.

Aspila, K. and A.S.Y. Chau. Quality Assurance and Certified Reference Materials. Essential Components for Environmental Research, Assessment and Management. Idem.

Chau, A.S.Y. and Y. Stokker. Modular Concepts in Method Development: An Example on the Development of a Multi-Class Procedure for 37 Organics in Sediment, idem.

Stokker, Y.D., K. Aspila and A.S.Y. Chau. The Development of Certified Reference Materials for Environmental Analyses. Presented at the 15th Annual Meeting of the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS), Boston, Massachusetts. October 30-November 4, 1988.

Study 84022: WQB, PPWB and Federal/Provincial Quality Assurance Programs

by

H. Alkema and A.S.Y. Chau

INTRODUCTION

Water Quality Branch (WQB) Quality Control Studies were initiated in the early 70's as part of the ongoing interregional quality control program (IRQC). Presently, some 40 parameters in four water samples are sent out bimonthly to five regional WQB laboratories. Each bimonthly study provides for quick data evaluations within weeks of reported data. Six QA reports are prepared each year.

For the Prairie Province Water Board (PPWB), a QA progrm was initiated in 1982 as an ongoing program because there was "a need to improve the compatability of federal and provincial water quality data being collected in interprovincial and international streams in Western Canada". At present, three provincial laboratories, Alberta, Saskatchewan and Manitoba, and two WQB laboratories participate. The IRQC QA program was expanded and modified to meet the PPWB objectives of assessing long term data comparability and to enhance in-house quality control programs in these provincial and federal laboratories.

In the latter part of this decade, under several Federal/ Provincial (FP) WQ agreements, five more laboratories were integrated with the IRQC-PPWB program. Laboratory results in these programs are tested in absolute terms with the use of certified reference waters. Generally, laboratories report their results in a timely manner and take corrective action when results are identified as anomalous.

RESULTS

Two reference waters for the PPWB long term assessment are being finalized and tested for stability.

An extensive computer program for automated evaluation of results is in the testing stage.

Twelve final reports on interlaboratory QA were sent out to laboratory heads and their managers.

Ninteen preliminary data evaluations were quickly sent out to lab heads to identify problem areas and to assist in their internal QC.

Three compiled reports, NWRI contributions, in English and French for the Federal-Provincial QA Program were completed and sent to program managers.

Study 84022 (continued)

Professionally supported the LRTAP program in its development of statistics and computer programs.

INTERLABORATORY QA FINAL REPORTS - FEDERAL-PROVINCIAL AND PPWB

	Date	Report No.	Study No.
1.	May 31, 1988	RAB 88-04/05	FP25 and 26, PP65 and 66
2.	Aug. 8, 1988	RAB 88-09/10	FP27 and 28, PP67 and 68
3.	Sept. 19, 1988	RAB 88-12/13	FP27 and 30, PP69 and 70
4.	Nov. 30, 1988	RAB 88-15/16	FP31 and 32, PP71 and 72
5.	Feb. 21, 1989	RAB 89-03/04	FP33 and 34, PP73 and 74
6.	Mar. 15, 1989	RAB 89-07/08	FP35 and 36, PP75 and 76

Publications

Alkema, H. 1988. Summary Report for FPQA Studies 19-30, July '87-June '88, for Inorganics in Surface Waters. NWRI Contribution 88-74.

Alkema, H. 1989. Summary Report for the Interlab. Prairie Provinces Quality Assurance Program Studies 61-72 (Sept. '87-Aug. '88) for Inorganic Constituents in Surface Waters. NWRI Contribution 89-86.

Alkema, H. 1989. Summary Report for Interlaboratory FP Quality Assurance Program Studies 21-32, (Sept. '87 to Aug. '88) for Inorganics in Surface Waters. NWRI Contribution 89-87.

Study 84023: LRTAP Interlaboratory Quality Control Studies

by

K.I. Aspila, N. Arafat and A.S.Y. Chau

INTRODUCTION

The Federal-Provincial Long Range Transport of Airborne Pollutants (LRTAP) programs involve approximately 60 different laboratories that generate data on soft surface waters and precipitation samples. Many millions of dollars are invested in the laboratory production and use of these data. If these data are to be merged or used to define trends or acidic impacts, then these data must be unbiased and precise. This project helps define data quality, assists laboratories in taking corrective action and provides assistance to network managers who plan to merge data bases.

Studies for this project include ten soft natural waters that are distributed to normally 55 to 65 laboratories. Analyses are requested for about 20 constituents. Results are analyzed for measurement bias using the non-parametric approach of Youden. Individual results that deviate significantly from interlaboratory medians are flagged either high or low. Studies are normally completed about eight weeks after distribution. Each laboratory is then provided a laboratory specific appraisal indicating their performance. Corrective action is suggested when performance is poor.

RESULTS

This project was initiated in 1982. Seventeen studies have been completed and for FY 88/89 three new ones, on major ions, nutrients and minerals were initiated. Study No. L18 and L19 are now complete. Study L20 is in progress and will be finalized by April 1989.

The external QA studies program continues to demonstrate that laboratories can improve their performance when they participate frequently in an effective peer group audit program (the round-robins) and have a management attuned to Quality Management, Quality Assurance and Quality Control concepts. Improvements are induced by the collective efforts of analysts, managers, active input by users of data, and frequent studies using a powerful design and interpretive technique (Youden's non-parametric technique).

Data assessment procedures are now heavily automated and utilize the mainframe Cyber computer to analyze the AQC data base (Sytem 2000 format). Two new procedure programs, one for creating precision functions and another for providing valuable data on charge balance and conductance ratios are being developed to further assist in defining laboratory performance.

Study 84023 (continued)

This LRTAP QA project has been remarkably effective and is accepted by the international community. Presentations to the US NAPAP (Austin, Texas and Denver, Colorado), the International Joint Commission (Windsor), Environment Canada (Ottawa) and the 3rd Chemical Congress (Toronto) has made this LRTAP QA project the focal point and primary instrument for new revenues (soft monies) for the QA project. There are now 102 different laboratories in the LRTAP QA studies.

Publications

Lawrence, J., K.I. Aspila and A.S.Y. Chau. 1988. Metrological Quality Assurance in Environmental Measurements. Proceedings of the 2nd Symposium on Metrological Assurance for Environmental Control, August 22-24, 1988, Helsinki University of Technology, Otaniemi, Espoo, Findland.

Aspila, K.I. 1989. Comparability Studies. Proceedings of the 2nd Ecological Quality Assurance Workshop, February 8-10, 1989, Environmental Research Centre, University of Nevada, Las Vegas, Nevada, U.S.A.

Presentations

Aspila, K.I. and A.S.Y. Chau. On Designing, Conducting and Evaluation of International and National Interlaboratory QA Studies. 3rd Chemical Congress, Toronto, June 5-11, 1988.

Lawrence, J., K.I. Aspila and A.S.Y. Chau. Metrological Quality Assurance in Environmental Measurements. 2nd Symposium on Metrological Assurance for Environmental Control, Keynote Lecture II, Helsinki University, Findland, August 22-24, 1988.

Aspila, K.I. Comparability Studies. Second Ecological QA Workshop, University of Nevada - Las Vegas, February 8-10, 1989.

Study 84024: IJC Quality Control Studies

by

K.I. Aspila, and A.S.Y. Chau

INTRODUCTION

Agencies that support the Canada-US Great Lakes Water Quality Agreement routinely generate data on many environmental substrates. These analytical data are made available to the institutions of the International Joint Commission (IJC). To assist the users of this data and to help assist laboratories in creating high quality data the Data Quality Work Group provides interlaboratory studies.

Studies are evaluated by the non-parametric technique of Youden and Wilcoxan. These techniques provide powerful methods to discern laboratory measurement bias. Individual results that deviate significantly from interlaboratory medians are also flagged high or low.

RESULTS

Study No. 56 - Metals in sediments was distributed to over 30 laboratories within the Great Lakes Basin. The study consisted of ten Great Lakes sediments and analyses were requested for approximately 20 metals. The results have clearly demonstrated that many laboratories failed to estimate "total" metals (e.g., Al, V, Fe, and Cr). The failure relates to inappropriate digestions that failed to recover the above metals. Other metals such as Cu, Pb, Zn, and Hg were well analyzed. Some problems appear for As and Se with results that are perhaps rather high. This matter requires further review.

Interlaboratory Study No. 62 - Phospnorus in water was distributed in the summer of 1988 and an evaluation report distributed in the fall.

Ampules of toxic organics suitable for direct GC analysis were partially prepared. Study No. 59 (ampuls) and Study No. 55 (toxic organics in sediments) were rescheduled.

Publications

Cheam, V., A.S.Y. Chau and W. Horn. 1988. National Interlaboratory Quality Control Study No. 35 - Trace metals in Sediments. Inland Waters Directorate Report Series No. 76.

Cheam, V., K.I. Aspila and A.S.Y. Chau. 1988. Analytical Reference Materials. VIII. Development and Certification of a New Great Lakes Sediment Reference Material for Eight Trace Metals. NWRI Contribution 88-71. Sci. of Total Environment (in press).

Study 84025: External QA for the Eulerian Model Field Study

by

K.I. Aspila and W.C. Li

INTRODUCTION

The governments of Canada, the United States and Ontario as well as the Electric Power Research Institute in America are, for a period of 24 months, collecting daily rainwater samples in their respective precipitation networks. Chemical analysis data from these four networks will be merged and the Eulerian model for atmospheric transport will be evaluated.

To assist managers of the Eulerian Model Field Study Program, the QA group in the Research and Application Branch has arranged to provide monthly interlaboratory performance evaluation studies. Participants in these studies include the four primary laboratories in the Eulerian program as well as several other laboratories (four or five). Each month these laboratories are provided ten precipitation samples for analysis of 14 specific constituents. Results are appraised for bias by the non-parametric method of Youden. Individual results that deviate significantly from interlaboratory medians are flagged high or flagged low.

RESULTS

The interlaboratory studies were implemented in July 1988 and studies have continued since on a monthly basis. Laboratory specific appraisals were mailed to each laboratory within four weeks of completing each study. Results of each study have been entered into a SYS2K data base management system for future retrieval and use.

Computer programs, using data base retrievals are being developed to create precision functions (essential for establishing criteria) and to address charge balance and conductance ratio.

Many dozens of precision functions were created and after careful review in the summer of 1989, will be used to establish improved criteria required to flag deviating results in interlaboratory studies.

Reports on studies E018L, E1, E2, E3, E019L, E4, E5, E6, E020L, E7, E8 and E9 were prepared and distributed to program and laboratory managers. Several studies revealed blunders that were serious, corrective actions were taken.

STUDY REPORTS

HYDRAULICS STUDIES PROJECT

Study 84032: Fluvial Engineering

by

P. Engel, B.G. Krishnappan, Y.L. Lau, P. Pelletier, K. Wiebe

INTRODUCTION

The work in this area focussed on developing a flow measuring technique for rivers carrying a heavy sediment load; examining the characteristics of composite weirs for small research watersheds, new technology for measuring streamflow; development of a calibration strategy for suspended sediment sampler calibrations; establishment of quality control criteria for discharge measurement under ice; and the development of large culvert gratings.

RESULTS

Milk River Flow Measuring Structure

Sand bed streams often present problems in the determination of the total volume of water passing a gauging station because they are subject to changes in bed roughness and bed elevations due to scour and deposition. As a result, stage-discharge relationships are often poorly defined and corrections based on frequent discharge measurements must be applied. The Milk River at Eastern Crossing, Montana, is an example of such a stream. The waters of the Milk River are shared by Canada and the United States in accordance with the Boundary Waters Treaty of 1909 and a 1921 order of the International Joint Commission. Satisfactory apportioning of the water requires that reliable discharge records are available at the Eastern Crossing gauge. Work is being completed on testing the final design of an artificial control to be used at Eastern Crossing. The final design is a flat-vee weir and tests so far indicate that this structure will operate satisfactorily under all but the most severe conditions.

A report based on tests completed last fiscal year was completed. Further tests on the better of the two structures initially tested were conducted and completed and a report has been prepared. In addition, a paper dealing with the last set of tests as well as the electro-magnetic method of total flow measurement was prepared jointly with RRB and WSC. The paper has been accepted for the CSCE Conference this summer in Saint John's, Nfld. The work was conducted in support of WRB, Calgary, Alberta.

Composite Weir

A theoretical and experimental study to investigate the behaviour of combined sharp crested rectangular and "V" notch weirs was completed and a report prepared. The work was in support of WRB, Guelph, Ontario.

Study 84032 (continued)

Acoustic Flowmeters

A final report on the "AFFRA" acoustic flowmeter has been completed. This report deals with the comparison of tests conducted in the towing tank and in a river near Ottawa. Tests were conducted on a second instrument called the Stork Servex Ultrasonic Flowmeter, designed in Holland. The tests were conducted in the towing tank. As part of these tests, the performance of the towing carriage was also examined. A report was prepared for the Stork flowmeter tests and the towing carriage evaluation, respectively. The work was done for WRB at headquarters in Ottawa.

Vertical Velocity Profiling

Work on a method to measure mean velocity for flows with irregular vertical velocity profiles has been started. Theoretical considerations have been completed and the preparation of the experiments is underway.

Discharge Measurement Under Ice

Work is underway on a long term study to establisn quality control criteria for discharge measurement under ice conditions. The study is the result of a research plan devised by the WRB R&D committee. The first phase, dealing with the examination of different types of current meter calibration equations and the effects of different types of meter suspensions is underway.

Measurement of Suspended Sediment

Work has begun on the development of a comprehensive strategy for the calibration of all suspended sediment samplers used by WRB across Canada. Measurement of suspended sediment load and recent emphasis on considering fine sediment fractions as a water quality parameter, make accurate sediment sampling more important than ever. A preliminary report has been completed jointly with WRB.

Large Culvert Gratings

Work was begun on the development of a standardized design of a grating for large culverts in urban catchments. The grating design will significantly reduce the risk of drowning at these structures during storm runoff events. The theoretical considerations are nearing completion. Experiments will be conducted as soon as possible.

Study 84032 (continued)

WRB Hydrometric R&D Management Committee

P. Engel is a member of this committee which dea's with the preparation of research and development priorities for the improvement of river discharge measurements under ice conditions.

Water Levels

The theoretical analysis on the performance of pressure transducers in measuring water levels is continuing. A report will be prepared as soon as possible. This work is being conducted for WRB in Ottawa.

Publications

Engel, P. 1988. Laboratory Tests of Artificial Control for the Milk River - Phase I. NWRI Contribution 89-90.

Engel, P., Y.L. Lau and M. Spitzer. 1989. Effect of Bed Level Fluctuations on Discharge Measurement in Alluvial Streams. NWRI Contribution 89-88. Paper accepted for CSCE Conference, Saint John's, Nfld.

Engel, P. and D. Doede. 1988. Stage Discharge Equations for Sharp Crested Composite Weirs. NWRI Contribution 88-75.

Engel, P. and E. Fast. 1989. Evaluation of the Stork Acoustic Flow Meter. Restricted Circulation Report.

Engel, P. 1989. Preliminary Examination of the Variability in the Towing Carriage Speed. NRWI Contribution 89-89.

Study 84033: Coastal Engineering

by

M.G. Skafel, C.T. Bishop, M.A. Donelan and J. Doering

INTRODUCTION

The work in the coastal engineering study this year is concentrated on wave-structure interaction, wave prediction, and Great Lakes water levels.

The wave-structure interaction work, partially funded by the Panel on Energy Research and Development focusses on the statistics of shallow water waves. The wave prediction work compares recommended techniques against field data. The water levels work provides background information for establishing water levels in the Great Lakes for structural design.

RESULTS

Shoaling Waves

This experiment was designed to look at the effects of wave shoaling on the joint probability density function (JPDF) of wave height and period; such information is required for the safe engineering design of coastal structures.

The field program, although initially beset with a full complement of teething troubles, produced several hours of excellent data. In the final storm of the experiment the waves were strongly shoaling between the tower in eight metres of water and the one at four metres. The tower closest to shore provided excellent data on waves in very shallow water (two metres).

In August 1988, Jay Doering arrived at NWRI to begin a NSERC post-doctoral fellowship. While at NWRI, Jay will be working with Mark Donelan, Michael Skafel and Craig Bishop on the analysis of Lake Ontario wave data from the Shoaling Waves experiment to determine the joint distribution of wave heights and periods in shoaling water depths. Subsequently he will be involved with laboratory tests of the same phenomenon. The results of this work will be of interest for the design of structures in shallow water.

The analysis of the field data collected during the 3-towers experiment has begun. The result indicate that the deep water JPDF of wave height and period is significantly modified by shoaling, as anticipated. The transformation of the JPDF is first characterized by a shift in the most probable wave period resulting from harmonic generation, then by a decrease in the expected wave height due to depth limited breaking. Work has also begun in the 103 m long wind-wave flume to conduct experiments to complement the field data. The lab experiments will examine the role of beach slope

Study 84033 (continued)

(which was, of course, fixed for the field study) in the transformation of the JPDF of wave height and period, and the change in the wave-induced forces arising from wave shoaling.

Wave Prediction

A paper entitled "Shore Protection Manual's Wave Prediction Reviewed" by Craig Bishop, Mark Donelan and Kimmo Kahma was submitted to and declined by the ASCE Journal of Waterway, Port, Coastal and Ocean Engineering. In this paper, revisions to simple wave prediction methods in the latest (1984) version of the Shore Protection Manual are shown to significantly overpredict wave height and period. We feel strongly in the merits of our paper, so after further revisions, we plan to submit it to another journal, Coastal Engineering, published by Elsevier.

Several one-dimensional wave models that have been adapted for computer application to determine wave climates, and a two-dimensional model, are being compared to numerous wave data sets, including some that have directional data. The analysis is continuing.

Great Lakes Water Levels

Work on the historical variation of Great Lakes water levels has been summarized and a paper submitted to the Journal of Great Lakes Research by Craig Bishop: "Historical Variation of Water Levels in Lakes Erie and Michigan-Huron". The major conclusion is that, over at least the last 400 years, climate-related variations in maximum mean annual water levels have probably not exceeded those measured on these lakes since A.D. 1819. A related paper is in preparation, co-authored by two archaeologists, to explain some effects of changes in water levels on Great Lakes archaeology. It is planned to submit this paper to a journal of archaeology before Christmas 1989.

Wave Forces on Pipelines

A series of experiments were completed, the goal of which was to shed light on the question of wave forces on pipelines in submarine trenches. As a result of discussions with oil company officials, pipe-trench configurations were selected that were representative, as far as possible, of those anticipated in the Beaufort Sea. Analysis of the results is continuing. In addition, flow visualization tests were conducted in a small wave flume to gain insight into the complexities of the flow close to the pipe. This work was partially funded by the Panel on Energy Research and Development.

Study 84033 (continued)

Publications

Bishop, C. 1988. Historical Variation of Water Levels in Lakes Erie and Michigan-Huron. NWRI Contribution 88-72.

Bishop, C.T. 1988. Water Levels. A lecture in: Coastal Engineering Basics, J.W. Kamphuis [Ed.]. Queen's University, Kingson, Ont., 32 p.

Bishop, C.T. 1988. Wave Prediction: A lecture in: Coastal Engineering Basics, J.W. Kamphuis [Ed.]. Queen's University, Kingston, 20 p.

Bishop, C.T. and M.A. Donelan. 1988. Waves and Wave Forecasting. In Civil Engineering Practice, Vol. 3, Geotechnical/Ocean Engineering, P.N. Cheremisinoff, N.P. Cheremisinoff and S.L. Cheng [Eds.]. Technomic Publishing Co., Inc., Lancaster, Pennsylvania, U.S.:653-695.

Bishop, C.T. and M.A. Donelan. 1989. Wave Prediction Models. In Coastal Modelling: Techniques and Applications, V.C. Lakhan [Ed.]. Elsevier Science Publishers (in press).

Skafel, M.G. and C.T. Bishop. 1989. Flow Visualization Around a Pipeline in a Trench. RAB TN-89-06.

Bishop C.T. and J.P. Coakley. 1989. Discussion of: Stratigraphy of the Sixteen Mile Creek Lagoon and Its Implications for Lake Ontario Water Levels, J.E. Flint, R.W. Dalrymple and J.J. Flint. Canadian Journal of Earth Sciences (in press).

Presentations

Bishop, C. Wave Prediction and Water Levels. Presented at Queen's University, Kingston, as a seminar to students in Coastal Engineering course.

Study 84034: Wave Mechanics

by

M.A. Donelan

INTRODUCTION

The work in this area includes: breaking waves; shallow water wave generation; SWADE. The breaking waves experiment is providing new information about the processes associated with deep water wave breaking, which have a bearing on the transfer of toxics at the air-water interface, and the velocity distribution, important in the safe design of offshore structures. A team from NWRI, NOAA/GLERL, WHOI, and AES are continuing to analyze shallow water wave data collected in Lake St. Clair with the aim of understanding more fully the effects of shallow water on wave growth. The Surface Waves Dynamics Experiment (SWADE) explores the spatial and temporal evolution of wind generated waves and the effects of fluxes at the interface.

RESULTS

Breaking Waves

Progress in this long term study was on two fronts: the analysis of directional spectra and the analysis of turbulence data from the drag spheres.

The directional spectrum analysis was focussed on cases in which a new wind sea was being generated in the presence of swell. There were three such occasions, in particular, the final storm of the experimental period, in which the wave heights often exceeded four metres. The principal new finding from this work is that there is a pronounced reduction of the wind sea when steep swell is present.

The directional spectrum analysis was refined further to include an iterative version of the Maximum Likelihood Method. The original method, although adequate for most of the wind sea spectrum, tended to broaden swell and lower frequency wind sea components. The iterative version adjusts for the non-negligible window width and, we believe, yields a faithful representation of the spreading of various wave components having wavelengths from 0.5 m to 150 m. A paper, entitled "Wave Directional Spectra in Mixed Seas" by I.K. Tsanis and M.A. Donelan, has been published in the Proceedings of the 2nd International Workshop on Wave Hindcasting and Forecasting sponsored by the Environmental Studies Revolving Fund.

The drag sphere work was intiially concerned with a comparison of these instruments with the Woods Hole acoustic current meter (BASS). The laboratory cross-calibration showed good agreement in a variety of flows with a mixture of oscillatory (waves) and steady (current) flows.

Study 84034 (continued)

Further investigation of wave-turbulence interaction via the drag sphere data was shelved awaiting the completion of the comparison between this instrument and the acoustic current meters of the Woods Hole Institute of Oceanography. The comparison is complete and we are gearing up for the final stage of the wave-turbulence interaction analysis and interpretation.

Analysis of the high frequency atmospheric turbulence has begun. The estimation of all the terms in the kinetic energy balance is possible from the data gathered during 1987. This will mark the first time that the turbulent kinetic energy budget has been fully explored in the surface bounday layer over water. The results will be potentially very valuable in putting the "dissipation method" of air-water flux estimation on firmer footing.

A short paper entitled "Air-Water Interaction Experiments in Lake Ontario by I.K. Tsanis and M.A. Donelan has been prepared for the CANCAM '89 conference.

SWADE

The principal activities in this large field experiment have been the preparation of the Gulf of Mexico tests and the scientific and fiscal planning of the whole experiment.

Staff of the Research Support Division have prepared our measurement system for the NDBC 3 m buoy and have hired a technologist to assist with this work. Technical Services staff have calibrated the anemometers in the towing tank and have completed an exploration of the flow and pressure disturbance around a model of the buoy in the wind-wave flume. These tests were required to determine the best placement of the various instruments on the buoy.

The planning of the Surface Wave Dynamics Experiment is well in hand. Five fixed-wing aircraft and one blimp will comprise the airborne data collection platforms during the three intensive periods in November 1990 (one week), January and February 1991 (two weeks each). The surface network, consisting of a spar buoy, two pitch/roll/heave buoys and about a dozen mean meteorological and wave rider buoys, will be operational throughout the six month (October 1990 to March 1991) phase of SWADE. The spar buoy has been refurbished and the design of its instrumentation and recording systems is underway. The prototype pitch/roll/heave buoy is undergoing tests in the Gulf of Mexico. The tests are comparisons with equivalent instrumentation on a Shell Oil production platform.

Study 84034 (continued)

The Gulf of Mexico tests, involving the calibration of the buoy systems against comparable tower mounted ones started in mid-January. The data from the Gulf of Mexico tests are to be recovered in early June. François Anctil, a Ph.D. student from the Civil Engineering Department of Laval University will spend four months (June through September) at CCIW analysing and interpreting these data.

Shallow Water Waves

The effects of shallow water on the development of the energy spectrum are being examined using suitable events recorded in Lake St. Clair. The evolution of the directional spectrum where there is an asymmetrical fetch and in the presence of a current is being examined using a maximum likelihood technique. Preliminary analysis has been completed in both cases.

The anomalous wave directions from Lake St. Clair during westerly winds appear to be due to refraction on the hydraulic current, which is concentrated near the western side of the lake. A more careful look at the directional distributions is warranted and preparations are being made to use the iterative Maximum Likelihood Method to help resolve the matter.

Publications

Kahma, K.K. and M.A. Donelan. 1988. A Laboratory Study of the Minimum Wind Speed for Wind-Wave Generation. J. Fluid Mechanics 192:339-364.

Graber, H.C., V.J. Cardone and M.A. Donelan. 1988. Global Ocean Wave Spectral Information from Space. Final report to the Physical Oceanography Program, Oceanic Processes Branch, U.S. National Aeronautics and Space Administration.

Presentations

Skafel, M.G., M.A. Donelan, H. Graber, P. Liu, D. Schwab and S. Venkatesh. Observations of Spectral Changes of Waves in Shoaling Water. Presented at the Canadian Meteorological and Oceanographic Society (CMOS) Meeting, Hamilton, Ontario, June 7-10, 1988.

Simons, T.J., W.M. Schertzer, M.G. Skafel and M.A. Donelan. Observations and Models of Wave-Induced Sediment Resuspension in Lake St. Clair. Presented at the CMOS Meeting, Hamilton, Ontario, June 7-10, 1988.

Tsanis, I.K. and M.A. Donelan. Directional Spectra of Waves in Changing Wind Conditions. Presented at the CMOS Meeting, Hamilton, Ontario, June 7-10, 1988.

Study 84034 (continued)

Presentations

Donelan, M.A., I.K. Tsanis, K.K. Kahma and N. Madsen. Wind-induced Pressure Measurements in Favourable and Adverse Winds. Presented at the CMOS Meeting Hamilton, Ontario, June 7-10, 1988.

Valdmanis, J., I.K. Tsanis and M.A. Donelan. Description of the "WAVES" Platform and Related Equipment. Poster Presentation at the CMOS Meeting, Hamilton, Ontario, June 7-10, 1988.

Donelan, M.A. 1989. The Science and Art of Wave Forecasting: Overview. Invited talk to the Symposium on Directional Ocean Wave Spectra. The Johns Hopkins University, Applied Physics Laboratory, April, 1989.

Merzi, N., I.K. Tsanis and M.A. Donelan. Observations of the Structure of Near-surface Turbulence in the Presence of Wind Waves. Presented at the Canadian Meteorological and Oceanographic Society (CMOS) Meeting, Hamilton, Ontario, June 7-10, 1988.

Merzi, N. and M.A. Donelan. Small-Scale Wave Breaking and Consequences for Gas Transfer. Presented at the CMOS Meeting, Hamilton, Ontario, June 7-10, 1988.

Study 84035: Transfer of Toxic Gases

by

M.A. Donelan and N. Merzi

INTRODUCTION

The work conducted in this study focusses on the physical processes that control the transfer rates of toxics between the water and the air. Adequate definition of these processes is important so that the pathways and mass balances of toxics in the environment can be computed.

RESULTS

Gas Transfer

The gas transfer flume is complete and has been shown to meet all its specifications including airtightness. It has been equipped with a fan capable of driving the wind in a closed circuit at centre line speeds up to 25 m/s or about 100 knots at the 10 m reference height. The water can be recirculated in either direction at speeds up to 50 cm/s and a hydraulically driven wave paddle can be used for regular or irregular waves. A speed control system was added to the recirculating water pump to avoid the heating caused by the original system of valve control on a constant speed centrifugal pump. The possibility of damage to the flume caused by sudden changes in atmospheric pressure has been avoided by the use of a pressure relief trap. Three automatic (micro-processor controlled) profiling systems have been added. These are designed to provide physical measurements (speed, temperature, humidity in air) at a variety of heights/depths in the air and water boundary layers. The instruments may be positioned vertically with a precision of 0.025 mm.

A series of measurements have been made of wave properties at three fetches and for various wind speeds. The first successful chemical tests have been conducted using chlorobenzene dissolved in the water phase. In these tests the flume was run open (that is, vented to atmosphere) and the rate of change of concentration of chlorobenzene in the water was monitored using a gas chromatograph. The results provide an accurate estimate of the evaporation exchange coefficient for this test substance. A program of measurements using other substances is being designed. The ultimate aim is to establish the dependence of air-water transfer properties on wave characteristics, water temperature, Reynolds number, Schmidt number and Henry's constant.

Publications

Merzi, N., E.A. Terray and M.A. Donelan. 1988. Energy Dissipation Just Beneath the Air-Water Interface and Consequences for Gas Transfer. NWRI Contribution 88-77.

Study 84035 (continued)

Presentations

Merzi, N., I.K. Tsanis and M.A. Donelan. Observations of the Structure of Near-Surface Turbulence in the Presence of Wind Waves. Presented at the Canadian Meteorological and Oceanographic Society (CMOS) Meeting, Hamilton, Ontario, June 7-10, 1988.

Merzi, N. and M.A. Donelan. Small-Scale Wave Breaking and Consequences for Gas Transfer. Presented at the CMOS Meeting, Hamilotn, Ontario, June7-10, 1988.

Study 84036: National Calibration Service

by

B. Taylor, D. Fekyt, C. Bil and B. Near

INTRODUCTION

This study encompasses NWRI's commitment to calibrate and repair all flow meters owned and used by Water Survey of Canada (Water Resources Branch, Environment Canada). This work is organized and conducted through the National Calibration Service.

In addition to the services provided to Water Survey of Canada, the National Calibration Service provides calibration and instrument repair services to outside clients on a cost-recovery basis.

RESULTS

A total of 161 requests have been actioned from Water Survey offices throughout the country and three universities, five provincial departments, three federal departments, two hydro utilities, three private companies and one USA agency. These requests resulted in a total of 839 calibrations for the fiscal year.

The tow carriage system underwent a detailed condition evaluation by an engineering consultant from Germany. Although the facility is considered to be in very good condition for its age, there are a number of maintenance requirements were recommended by the consultant.

A request for funding the tow carriage overhaul was submitted in the main estimates for 1989/90.

Study 84037: Hydraulics and Sedimentology Research Support and Facility Maintenance

by

W.B. Taylor, Technical Services Staff, J.P. Coakley and M.G. Skafel

INTRODUCTION

This study provides for specialized technical support staff and fully operational laboratories and equipment for hydraulics, and geotechnical research and related activities.

The human resources of the Technical Services Section were fully utilized to complete NWRI mission research as well as providing resources to cost-recovery projects (see Study 84038).

RESULTS

Staff were assigned to studies on a task-by-task basis and records of time utilization were maintained. All costs related to individual studies were assumed by the project being supported. A large area of the Hydraulics Laboratory has been allocated to new annular flumes being built for RRB.

The Sedimentology Lab staff have caught up on the large backlog of sediment size analysis, over 1600 samples were processed, and work on settling tube development is in progress. J.P. Coakley (LRB) is providing scientific advise on methods development.

The Sedimentology Lab is now operating and maintaining the large common-user freeze dryer. This service will be on full cost-recovery next fiscal year.

The Hydraulics Laboratory's capital replacement plan was updated and a request for funding will be submitted next fiscal year.

Study 84038: Applied Research and Engineering

by

Scientific, Engineering and Technical Staff of
Research and Applications Branch and Rivers Research Branch

INTRODUCTION

The work in this area is entirely driven by the requests of outside clients. The Windermere Basin Study was started last fiscal year and completed this year. The other projects were all started this year or were estimated this year. The two that have been estimated but have not been started are awaiting approval from the client to proceed.

RESULTS

Mar-Land Engineering - Windermere Basin Model Study

A physical model study was conducted to determine the hydraulic properties of the re-designed Windermere Basin in the south end of Hamilton Harbour, as well as evaluate the effect of different flow conditioning schemes on the hydraulic efficiency of the new basin. The basin was modelled using the Froudian law of similarity with a horizontal scale of 1:60 and a vertical scale of 1:15, resulting in a distorted model. A dye was used to identify flow patterns. Flow retention times were determined using a neutrally buoyant salt solution as a tracer. Several flow conditioning schemes were examined, including spur dikes, a weir at the outflow end of the basin, a flow deflector at the entrance of the basin and a sediment trap. An extensive report was prepared for the client Mar-Land Engineering Ltd., Consulting Engineers, Markham, Ontario.

Lafarge Construction - Erosion Control Products

A brief study was conducted on an erosion control product fabricated by Lafarge Construction Materials of Calgary, Alberta. This company is manufacturing a down-sized version of the Mini-Slab as produced by OAKS Precast Industries of Guelph, Ontario. The Mini-Slab is a slotted concrete block with horizontal interlock. The blocks are linked together to form a matrix which is used in drainage channels on surfaces subject to erosion. An analysis was conducted to determine the effect of down-sizing on their stability in fast open channel flow. The results showed that a size reduction of 12.5% resulted in a loss of stability of no more than 7%. A report was prepared for the client.

Shawinigan-Lavalin Inc. - Dam Break Surges

Extensive laboratory tests will be conducted in a specially designed flume and ancillary equipment to obtain experimental data of dam break surges with erosion effects for the verification of a numerical model. A detailed cost estimate was prepared for Shawinigan-Lavalin Inc.

Study 84038 (continued)

Sandwell Swan Wooster Inc. - Port Colborne Breakwater Study

At the request of Sandwell Swan Wooster Inc., the NWRI Hydraulics Laboratory conducted physical hydraulic model tests of breakwaters at Port Colborne, Ontario. As part of the engineering preparation for the tests, wave generation and analysis software, known as GEDAP, that was developed at NRC was made operational in our Hydraulics Laboratory. This enables staff to offer more sophisticated wave generation and analysis options to future clients, and, of course, accommodate their own research needs.

Defence Research Establishment - Offshore Research Tower

The Offshore Research Tower was rented to the Defence Research Establishment, Ottawa, for the month of November, so that they can conduct radar transmission tests.

Region of York - Parshall Flume

At the request of the Region of York, a model of a Parshall Flume, has been constructed in the laboratory to conduct calibration tests. The prototype is presently operating in a sewer, measuring discharge.

Acres International Ltd. - Northumberland Strait Crossing

A preliminary cost estimate was prepared for Acres International Ltd. to conduct hydraulic model tests in the NWRI Hydraulics Lab to determine suitable armour protection against scour by waves or currents for bridge piers at the proposed Northumberland Strait crossing.

Newsletter - Hydraulics Laboratory

A newsletter from the Hydraulics Laboratory was prepared, desktop-published and mailed in November, 1988. It contained information on research news, client studies, developments in facilities and equipment, and staff news. The Newsletter is distributed to approximately 600 readers made up of research associates in universities and government departments, consulting engineers, other potential clients, and other interested parties in physical hydraulics research. Newsletter #2 will be mailed in May 1989.

Atria Engineering Hydraulics Inc. - Trench Infilling

The small wave flume was rented to Atria for some qualitative tests of infilling rates of trenches for pipelines. The study was conducted by Atria staff with assistance from NWRI staff.

Study 84038 (continued)

RWDI Inc. - Hydraulic Model Study

Information was supplied to RWDI Inc. in order for them to prepare a proposal which would include a hydraulic model study in the NWRI Hydraulics Lab. The study would examine waves in the approach channels to a coastal power station, as well as pipeflow hydraulics in the pumphouse.

Hayward Gordon Limited - Large Observation Tank

The labortory's large observation tank was rented to Hayward Gordon Limited for testing a prototype mixer for the rubber manufacturing industry. The 5m long unit, weighing 5200 kg, and driven by a 60 hp electric motor was by far the largest device ever put into the tank. The test was successful.

The following summarizes the cost recovery activities for the fiscal year 1988/89.

Client	Project	Status	Study Leader
Mar-Land Engineering	Windermere Basin Model	complete	P. Engel
York Region	Parshall Flume Cal.	in progress	J. Marsalek
B.C. MOE	Parshall Flume Cal.	complete	J. Marsalek
Shawinigan-Lavalin	Dambreak Model	proposal	P. Engel
Sandwell SW	Marina Breakwater Tests	complete	Bishop/Skafel
Gore and Storrie	Manhole Section Calib.	complete	J. Marsalek
Lafarge Construction	Mini-Slab Data Analysis	complete	P. Engel
DREO	Offshore Research Tower rental	proposed	M. Skafel
Acres	Northumberland Strait	proposed	Skafel/ Bishop/Engel
RWG	Multiple Pump Intake Physical Model	proposed	Engel/Bishop

Study 84038 (continued)

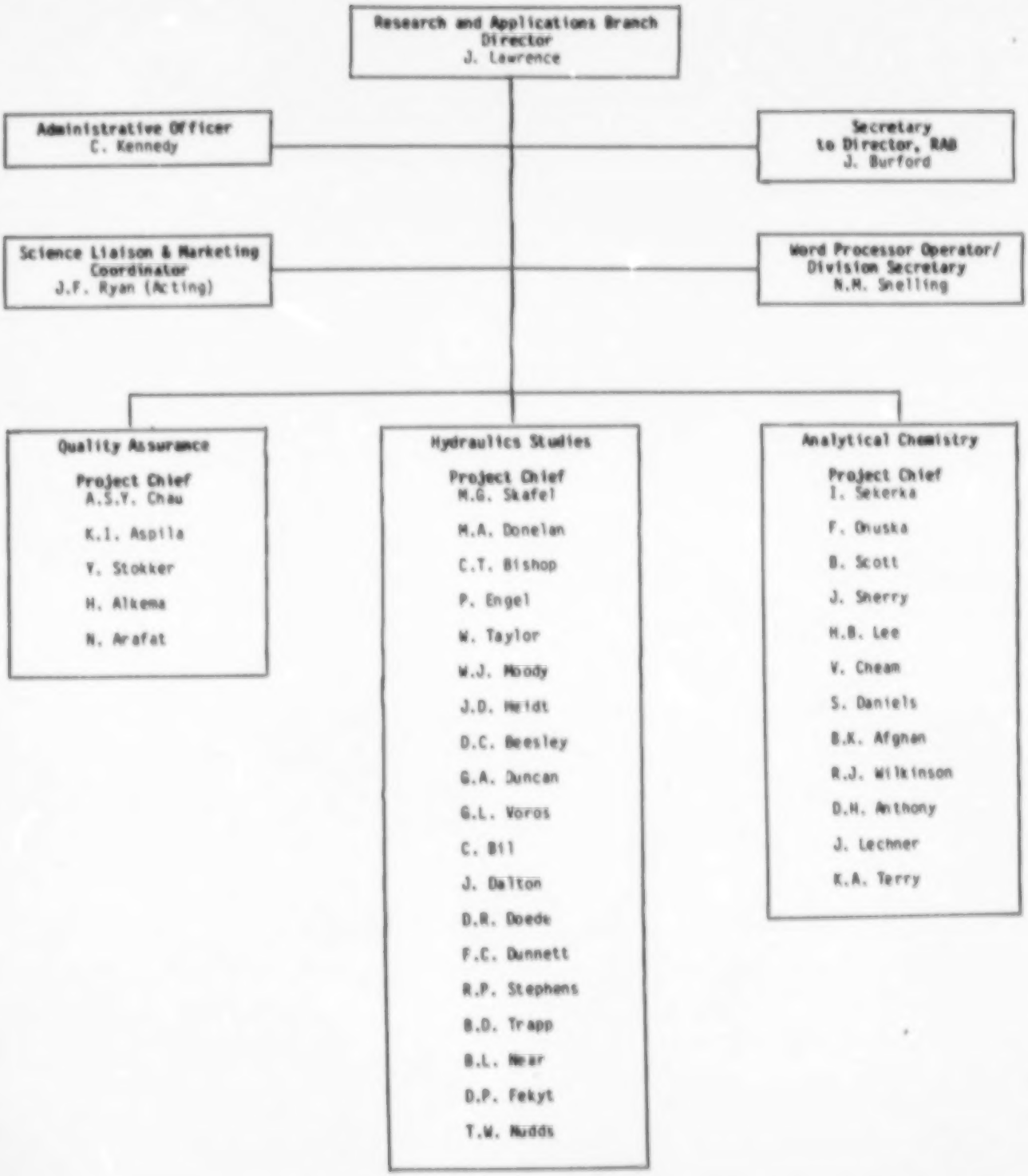
Publications

Engel, P. 1988. Hydraulic Model Study of the Windermere Basin. Restricted Circulation Report.

Engel, P. 1988. The Effect of Down-Sizing of Mini-Slabs on Critical Velocity. Restricted Circulation Report.

APPENDIX

ORGANIZATION CHART



RESEARCH AND APPLICATIONS BRANCH

Director's Office

Director

J. Lawrence, B.Sc., Ph.D. (Bristol)
Analytical Chemistry and Quality Assurance

Science Liason Coordinator

J.F. Ryan (Acting)

Secretaries

Ms. Joyce Burford
Ms. Norma Snelling

Administrative Officer

Ms. Colleen Kennedy

ANALYTICAL CHEMISTRY RESEARCH

STAFF LIST

Project Chief

I. Sekerka, RNC., RNDr. (Prague), CSc. (Ostrava)
Electroanalytical Chemistry and Flow Injection Analysis

Project Scientists

F.I. Onuska, P.Chem.Eng. (Bratislava), M.Sc. (Prague), CSc. (Brno), Ph.D. (Waterloo)
Gas Chromatography, GC/MS and Separation Techniques

J.P. Sherry, B.Sc., Ph.D. (N.U.I.)
Mycology and Radioimmunoassay

H.B. Lee, B.Sc. (Hong Kong), Ph.D. (McMaster)
Gas Chromatography, GC/MSD and High Pressure Liquid Chromatography

B.F. Scott, B.Sc., M.A., Ph.D. (Toronto)
High Pressure Liquid Chromatography and Gas Chromatography

V.F. Cheam; B.S., Ph.D. (Oklahoma)
Ion-Chromatography and Atomic Spectroscopy

S. Daniels, B.S., M.S. (Louisiana), Ph.D. (Waterloo)
Electrokinetic Methods

B.K. Afghan, B.Sc. (Sind), D.I.C. (I.C., London), Ph.D. (London)
Liquid Chromatography

Research Technologists

R.J. Wilkinson
J.F. Lechner
D.H. Anthony, B.Sc. (McMaster)
K.A. Terry

QUALITY ASSURANCE

STAFF LIST

Project Chief

A.S.Y. Chau, B.Sc. (British Columbia), M.Sc. (Carleton)
Reference Materials and Quality Assurance

Project Scientists

H. Alkema, B.T. (Ryerson)
Federal/Provincial QA Program and Preservation

K.I. Aspila, B.Sc., M.Sc. (Carleton)
Quality Assurance

Y. Stokker, B.Sc. (Guelph), M.Sc. (Queen's)
Organic Method Development and Quality Assurance

Technologists

N. Arafat, B.Sc. (Waterloo)

HYDRAULICS STUDIES

STAFF LIST

Project Chief

M.G. Skafel, B.A., B.E., M.Sc. (Saskatchewan), Ph.D. (Cambridge), P.Eng.
Coastal Engineering

Project Scientists

C.T. Bishop, B.Sc., M.Sc. (Queen's), P.Eng.
Coastal Engineering

M.A. Donelan, B.Eng. (McGill), Ph.D. (B.C.)
Air-Water Interaction

P. Engel, B.A.Sc., M.A.Sc. (Waterloo), P.Eng.
Fluvial Engineering

Technologists

W.B. Taylor, Head, Technical Services Section

D. Beesley
C. Bil
J. Dalton
D. Doede
G. Duncan
F. Dunnett
D. Fekyt
J. Heidt
W. Moody
B. Near
T. Nudds
R. Stephens
B. Trapp
G. Voros